Flux and Rejection of Amine Using Membrane Process

by

Fathin Fathiha Ahmad

Dissertation submitted in partial fulfillment of
the requirements for the
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CERTIFICATION OF APPROVAL

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Approved by,

_____
(Assoc. Prof. Dr. Hilmi Mukhtar)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

June 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

FATHIN FATHIHA BT AHMAD

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Praise to God and thanks to Him for His plan towards me to carry on with this Final Year Project and completed successfully on time given without any major difficulties. I dedicate a special thanks to my parents and family for their support and encouragement towards me all this while in completing this project.

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Hopefully this report will give more understanding and knowledge for all the readers about the separation behaviour of amine, TEA by using membrane and the information gained from this project.

ABSTRACT

Aliphatic amines are widely used as raw materials in much industrial process, and very harmful chemical species constituting the focus of many environmental concerns. The objectives of this project are to study the separation behavior of amine from natural gas purification plant using membrane process, to investigate and study the factors that affecting the performing of separation amine. Amine chose in this project is Triethanolamine, TEA, which is a tertiary amine. In refinery gas treatment plant, amine absorbed acid gas components, hydrogen sulfide, H_2S and carbon dioxide, CO_2. H_2S and CO_2 are absorbed by the amine solution and the sweet gas leaves the absorber. Once stripped of contaminants, amine solution is reused.

A suggested method to study the separation of amine is through membrane separation. The factors affecting the membrane performance are operating pressure, feed concentration, temperature, pH, concentration and cross flow velocity. Membrane performance is analyzed based on the flux or rejection/ separation of amine. Generally, the separation of amine from wastewater using membrane system could be implemented for industrial applications but with several modifications of transport and parameters that had to be studied further in order to achieve optimum results in the industry application.

The effect of different parameters in membrane performance will be observed and evaluated. The acquired results will be used for comparison of the importance of experimental parameters in optimization of amines separation. The flux was increased as operating pressure increased. Similarly, flux increased as velocity increased. CA202 has highest flux (104L/m^2.h), followed by AFC40 and lowest was AFC99 (0.5L/m^2.h). From the analysis and feasibility study, the membrane separation process is a good separation method in water treatment. AFC99 showed better result than AFC40 and CA202. AFC99 almost removed 89% of amine, compared to AFC40, 69% and CA202 49%. Further analysis need to be done by using several methods, techniques and other considerations should be taken in order to achieve the project objectives.

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CHAPTER 1

1.0 INTRODUCTION

1.1 Research Background

The current peak oil production causes more important role of natural gas in energy industry. Natural gas has been predicted to be the fastest growing fuel of world primary energy consumption. However, the crude natural gas often consist of carbon dioxide, CO_2 and hydrogen sulfide, H_2S that commonly known as sour gases. The high concentration of these gases in natural gas is undesirable and has to be removed (Furhacker et al., 2003). The gases cause corrosion, reduce the heating value and thus the sales value of the gas. Thus, to overcome this problem, technology for removal of CO_2 and H_2S has been applied and commonly called sweetening process.

In sweetening process, amine based absorption process has become the major existence technology (Arnold and Steward, 1988). Common amines that been used are Diethanolamine, DEA, Methyldiethanolamine, MDEA and Trietanolamine, TEA. In this project, TEA was selected as subject in research conducted. The properties of amine were attached in APPENDIX A. The source of amine to wastewater was from flushing of the absorption column in sweetening process. Also, the managed and unmanaged waste streams may be composed of spent amines, sludge from process unit tank bottoms, and the process system filters. Unmanaged waste streams, particularly spills during changeover operations (the process of exchanging spent chemical for fresh chemical), and may be include fresh amine (Sorensen, 1999).

Researched have been conducted on various methods and possibilities of separation as an innovative approach of amine separation that optimizes costs, space, safety, production and control. Amine removal can be grouped into physical, chemical or biological process. The conventional approaches in treating amine wastewater are by chemical and physical processes, such as adsorption, stripping, membrane filtration, electro dialysis and chemical oxidation (Tomei et al., 2003). These processes guarantee high removal efficiencies, but the first four approaches have the main drawback as they

do not provide a real degradation of the compounds but only transfer from a diluted to a concentrated stream. On the other hand, chemical oxidation could produce intermediate that has similar toxic level to the original substance. Therefore, chemical process usually required further treatment.

Biological treatment is an alternative and promising approach that could provide complete biodegradation of the compound with low investment and operation cost (Tomei et al., 2003). Another successful work has been published by Lai and Shieh (1996), using batch system via nitrate respiration for treatment of amine. Instead of biological suspended system, treatment of amine was also successfully treated in biological fluidized bed (BFB) (Shieh and Tsao, 2002).

The current approach to solve the environmental pollution from sweetening process was conducted by reuse and treatment of wastewater. The amine wastewater is recommended to be separated using membrane processes such as nanofiltration (NF) and reverse osmosis (RO) (Isa et al., 2005). The membrane offers a complete barrier to suspended solid and yield higher quality effluent (Visvanathan et al., 2000; Stephenson et al., 2000; Judd et al., 2003; Roest et al., 2005).

1.2 Problem Statement

Acid gas removal using amine is well understood because it has been widely used in oil and gas industry. Due to amine's ability to increase chemical oxygen demand, COD level of the effluent, separation of amine from the discharge water is a must-done-task. Membrane is rated for suitability as an application filter based on the criteria such as pore size and morphology, hydrophilicity, chlorine resistance, chemical resistance, pH range tolerance, temperature and pressure tolerance, permeability, stability of the pore structure, clean ability, fouling resistance and consistency and quality of the membrane.

Apart from other separation industries, membrane process is widely used in water purification industry or wastewater treatment plant to obviate suspended and dissolved solids, heavy metals and other kind of impurities from the water stream. The use of membrane to remove amines from the effluent water is still devoid. Therefore, this unprecedented study will highlight the feasibility of using membrane process to separate amines from wastewater.

1.3 Objectives and Scope of Study

Objectives in this project are;

1. To study separation behavior of artificial amine in wastewater by using membrane process
2. To study factors that influences the separation of amine using membrane processes
3. To evaluate membrane performance to reach the optimum separation of wastewater containing amine
4. To compare the performance of membrane types

The study covered all the research of the properties of amine and the performance of amine separation by using Membrane Test Unit in laboratory. The performance of membrane is to prove the ability of membrane for separation of wastewater containing amine. The test was mainly objected to evaluate filtration performance in term of flux and rejection. The system was compared in term of removal efficiency and the ability to

withstand any changes of feed concentration. Furthermore, the pattern on membrane filtration flux and rejection were monitored.

1.4 Relevancy of the Project

Amine in wastewater can not be separated easily due to its properties and have the ability to increase COD level of the effluent. The use of membrane to remove amine from effluent water is still devoid. Therefore, this unprecedented study will highlight the feasibility of using membrane process to separate amine form effluent water prior to discharge and environmental quality requirement.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1 Industrial Wastewater

Wastewater is defined as combination of liquid and water that carries wastes that were removed from residences, institutions and industry, together with such ground water, surface water and storm water (precipitation) (Metcalf and Eddy, 1991; Terbutt, 1998). When untreated wastewater is allowed to accumulate, the decomposition of organic materials leads the production of large quantities of malodorous gases. Wastewater also usually contains numerous pathogenic or disease-causing microorganisms. The nutrient rich wastewater that enter the aqueous ecosystem leads to euthropication, which still because oxygen depletion, it is also toxic to the aquatic life and responsible to methemoglobinemia when it is contaminated to drinking water.

2.2 Wastewater Characteristic

Industrial wastewater is characterized in term of physical, chemical and biological constituent. The important physical properties are color, odor and solid content. The chemical constituent may contain of organics such as carbohydrates, phenol, pesticides, etc; gases such as H_2S, methane and oxygen; and inorganic such as alkalinity, heavy metals, nitrogen, pH and others. The biological constituent may contain of animals, protista, virus, etc (Metcalf and Eddy, 1991; Benefield, 1980; Grady et al., 2001).

One of the important wastewater constituent is organic chemicals. This constituent has become one of the important concerns in determining the quality of wastewater. The organic chemicals usually are not specific and consist of mixture of many different carbonaceous materials. As a result, test for the organic content of such wastewaters are not specific. The two most common tests are biochemical oxygen demand, BOD and chemical oxygen demand, COD.

Wastewater treatments were first developed in response to the concern for public health and adverse conditions caused by the discharge of wastewater to the environment (Metcalf and Eddy, 1991). The objective of the process is to remove suspended and floatable materials, treatment of biodegradable organics and elimination of pathogenic organisms. Based on Environmental Quality Acts 1974, no person shall discharge effluent, analyzed in accordance with regulation, which contains substances in concentrations greater than those specified in parameter limits. The effluent discharged into any inland waters is categorized into two standards which are;

1. **Standard A** – the parameters shown in third column in the table below into any inland waters within the catchment areas specified in Malaysia. The catchment areas referred to in this regulation shall be the area upstream of surface or above sub-surface water supply intakes, for the purpose of human consumption including drinking.
2. **Standard B** – the parameters shown in forth column in the table below, into any other inland waters.

TABLE 2-1: Parameter Limits of Effluent of Standard A and B (*EQA, 1974*)

Parameter	*Unit*	*Standard*	
		A	B
1. Temperature	°C	40	40
2. pH value	-	6.0-9.0	5.5-9.0
3. BOD at 20°C	mg/L	20	50
4. COD	mg/L	50	100
5. Suspended solids	mg/L	50	100
6. Phenol	mg/L	0.001	1.0
7. Free chlorine	mg/L	1.0	2.0
8. Sulphide	mg/L	0.50	0.05
9. Oil and grease	mg/L	Not Detectable	10.0

2.2.1 Temperature

Temperature is basically important for its effect on the other properties, for example, speeding up of chemical reactions, reduction in solubility of gases, amplification of taste and odors and others (Terbutt, 1998). For the most part, temperature is not a critical issue below 37°C if wastewater is to receive biological treatment. Low temperature operations in northern climates can resulted in very low winter temperature and slow reaction rates for both biological and chemical treatment systems. Increased viscosity of wastewater at low temperature makes solid separation more difficult. Efforts are generally made to keep operating temperature between 10 and 30°C if possible (Davis, Berner, 2004).

2.2.2 pH Value

Wastewater should have the pH values between 6 and 9 for minimum impact on the environment. Wastewater with pH values less than 6 will tend to be corrosive as a result of the excess hydrogen ions. On the other hand, raising the pH above 9 will cause the metal ions to precipitate as carbonates or as hydroxides at higher pH levels. Alkalinity is important in keeping pH values at the right level. It is important to have adequate alkalinity to neutralize the acid waste components as well as those formed by partial metabolism of organics (Davis, Berner, 2004). pH value is satisfied by below formula with molar concentration of H^+ (Terbutt, 1998, p15);

$$pH= -\log_{10}[H^+]=\log_{10}(1/[H^+]) \tag{2.1}$$

2.2.3 BOD

Biochemical oxygen demand, BOD is the quantity of oxygen utilized by a mixed population of microorganism in the aerobic oxidation (of the organic matter in a sample of wastewater) at a temperature of 20°C ±1°C (Hammer, Hammer Jr., 2001). BOD is used to define strength of a municipal or organic industrial wastewater and to determine the relative oxygen requirements to treated effluents and polluted waters. Basically, COD value is higher than BOD value because chemical oxygen decomposes nonbiodegradable organic matter, and the standard BOD test measures only the oxygen used in metabolizing the organic matter for five days.

2.2.4 COD

Chemical oxygen demand, COD is to characterize the organic strength of wastewaters and pollution of natural waters. The test measures the amount of oxygen required for chemical oxidation of organic matter to carbon dioxide and water (Hammer, Hammer Jr., 2001). The impact of an effluent on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD permits the way of measuring the amount of organic waste in wastewater or effluent water. The higher the COD value, the more the organic wastes are, hence, the higher the amount of dissolve oxygen needed by the bacteria to break down the organic waste in the effluent. Normally, oxygen is not a very soluble gas in water, thus, dissolved oxygen concentration in wastewater is very low. As a result, higher COD will resort to anaerobic bacteria scrounging for and rip the bounded oxygen from water molecule for their biological activities. This process requires longer time and more energy needed by the anaerobic bacteria to digest the organic matter (Afdzal, Amiruddin, Azman).

2.2.5 Wastewater from Natural Gas Purification Plant

The consumptions of natural gas have been estimated to increase, however the production of natural gas often consists of sour gases which are CO_2 and H_2S. These gases act as pollutants in significant amount and have to be removed because they cause corrosion, reduce the heating value and the sales value of the gas (Arnold and Stewart, 1988). The amine based absorption for removal of sour gases from natural gas sweetening process has become the major existing technology. The amine solution, in combination with anti corrosives agent are used to adsorb acid gases in sweetening process. The used amine is recalcitrant waste that requires treatment before disposal (Arnold and Stewart, 1988).

2.3 Source of Amine to Wastewater

Stringent quality standards require amine to be removed from such vapor stream prior to its being vented to the atmosphere. From the FIGURE 2.3, the amine unit, the section is processed to remove gas H_2S from gas by amine. Gas is contacted with lean amine solution in the absorber. The amine absorbs the H_2S and some of the CO_2. The treated gas is sent to the thermal oxidizer where residual H_2S is converted to SO_2 before discharge to atmosphere. The rich amine is sent to the regenerator after being heated in the Lean/Rich exchanger by the hot lean amine from the bottom of the regenerator. In the regenerator, the acid gases are released from solution by heating the solution in the reboiler. The overhead from the regenerator is cooled and the condensate returned to the column. The cooled, water saturated, acid gas is recycled to the Claus unit where H_2S is further treat to recover more purity of sulphur. The hot amine is cooled firstly by heating the rich solution and then in the lean amine cooler before entering the absorber. Small percentage of amine carryover to sulphur plant is then been disposed to environment.

The wastewater from sweetening process unit comes from several sources. The major quantity of wastewater is produced during process turn-around (Isa et al., 2006) and small quantity is produced incidentally during the process operation. The sources of wastewater for amine system during operations are from;

i. Amine include in reclaimer
ii. Excessive amine carry over at the absorber
iii. Degraded amine that was removed through reclaimer
iv. Amine included in filter cake that was exposed to environment during backwashing
v. Liquid hydrocarbon from oil-gas separation
vi. Several others sources such as water used to wash the vessel and other equipments (heat exchanger, pumps), valve leakage and operational upset
vii. Managed and unmanaged waste streams may be composed of spent amine, particularly spills during changeover operations (the process of exchanging spent chemical for fresh chemical), and may be include fresh amine

(Furhacker et al., 2003; Arnold and Steward, 1988; Lai and Shieh, 1996)

This wastewater is introduced to main wastewater stream and treated at existing wastewater treatment plant (WWTP). Wastewater from sweetening process is characterized by up to 52,000 ppm COD (Anis, 2005), and in certain cases can reach of 350,000 ppm COD (Isa et al., 2006).

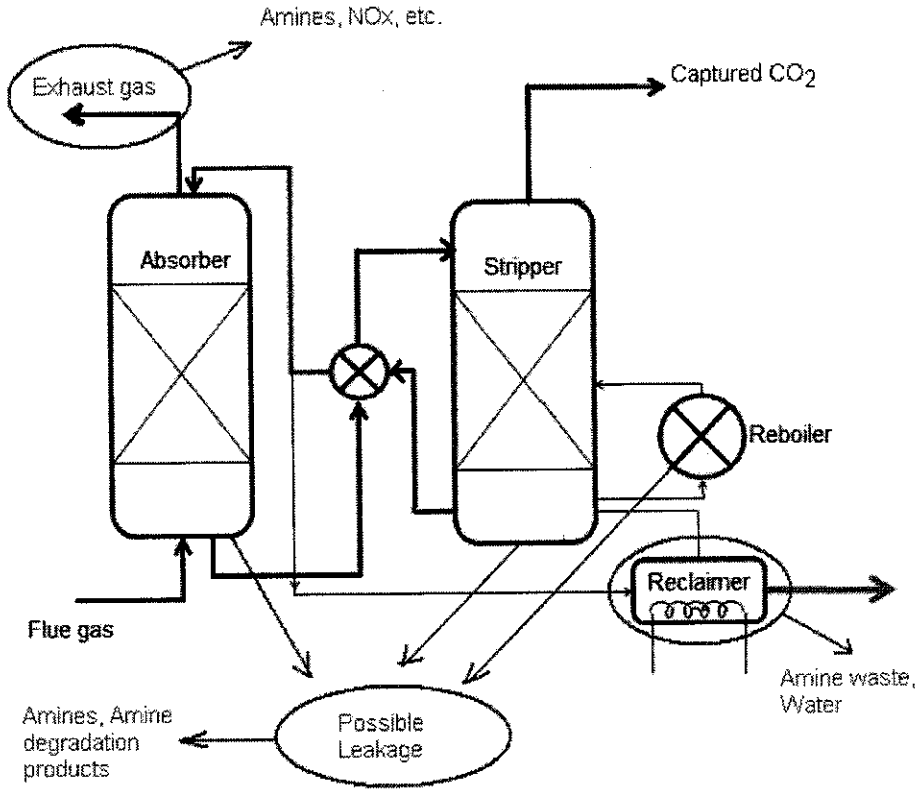

FIGURE 2-1: The possible emission sources of amines and degradation products for the CO_2 capture process (Jon Hovland, 2009).

2.4 Technology to Separate Amine

The invention relates to a method of removing amine from aqueous solution thereof, in particular from wastewater. Such amine solutions constitute a problem of waste disposal, because no method has been known, by which amine can be removed from wastewater in high yields and in a simple manner. Many amines are known to be toxic and difficult to degrade (Lenzing, 1991). Spent amine is difficult to biodegrade in wastewater plants and must be handled in accordance with rules and regulations for hazardous waste handling.

The current approach to solve the environmental pollution from sweetening process is conducted by reuse and treatment of wastewater. The amine wastewater is recommended to be recovered using membrane processes such as nanofiltration (NF) and reverse osmosis (RO) (Isa et al., 2005). Several processes have been developed for removal of amine from wastewater. Researched have been conducted on various methods and possibilities of separation as an innovative approach to amine separation that optimizes costs, space, safety, production and control. Amine removal can be grouped into physical, chemical or biological process.

2.4.1 Physical and Chemical Process

The conventional approaches in treating amine wastewater are by chemical and physical processes, such as adsorption, stripping, membrane filtration, electro dialysis and chemical oxidation (Tomei et al., 2003). These processes guarantee high removal efficiencies, but the first four approaches have the main drawback as they do not provide a real degradation of the compounds but only transfer from a diluted to a concentrated stream. On the other hand, chemical oxidation could produce intermediate that has similar toxic level to the original substance. Therefore, chemical process is usually required further treatment.

2.4.2 Biological Treatment

Biological treatment is an alternative and promising approach that could provide complete biodegradation of the compound with low investment and operation cost (Tomei et al., 2003). Conventional treatment system has been successfully applied to treat methyldiethanolamine, MDEA contaminated wastewater from sweetening process (Furhacker et al., 2003) with removal efficiency of more than 96% based on total organic carbon, TOC measurement. Another successful work has been published by Lai and Shieh (1996), using batch system via nitrate respiration for treatment of amine. The various nitrogen compound that majority consists of ethanolamine was also successfully biodegraded in membrane bioreactor (Chen et al., 2003). Instead of biological suspended system, treatment of amine was also successfully treated in biological fluidized bed (BFB) (Shieh and Tsao, 2002).

In biological process, wastewater is degraded in bioreactor by microorganism to produce the new cell (biomass) and more stable compounds. The biomass has to be separated to achieve a good effluent quality. In activated sludge process, biomass is separated in bioreactor by operating the system sequentially. For this purpose, the system consists of fill, react, settle, decant and idle phase. This process is commonly known as sequence batch reactor, SBR.

2.5 Amine in Wastewater

The process of refinery, amine in aqueous solution been used in capturing CO_2 on large scale. In such operations, emissions of amine occur through the cleaned gas, as degraded solvent and as accidental spills. It is thus important that the chemicals used have low or no environmental effects (Huagmo et al, 2009). Standard ecotoxicity and biodegradability have been used to determine the environmental risk. Some of the solvents used for carbon capture, have been shown to have low biodegradability. The tertiary amine which has been tested does not degrade easily. It is expected that that primary and secondary amine are more degradable than tertiary amines and compounds containing quaternary carbon (Huagmo et al, 2009).

Treatment plants received process water effluents from oil and gas industry and contained high potential for the formation of amine. The occurrence and removal of amine within wastewater treatment processes have been investigated intensively. On account of high production quantities and manifold applications in industry, amine is identified in industrial wastewater and municipal sewage.

Environmental partitioning is influenced by substituent as well as by number of carbon atoms and the amine structure of the carbon skeleton. The presence of amino group causes a higher boiling point, higher water solubility and a higher mobility in the water cycle in comparison with hydrocarbons. However, that depending on the milieu parameters, the amino group can also reduces the mobility of a molecule by specific interactions with solids via covalent bonding to carbonyl moieties or cation exchange. The volatility of amine in aqueous is in most cases relatively low. Hence, the atmosphere is hardly considered here.

FIGURE 2-2 summarizes important pathways of amine entering the environment. Both direct inputs via industrial waste and formation from precursors have to be considered (Jekel, Reemtsma, 2006). Amines are generally can cause problematic to human because of odor and taste impairment even at very low concentrations.

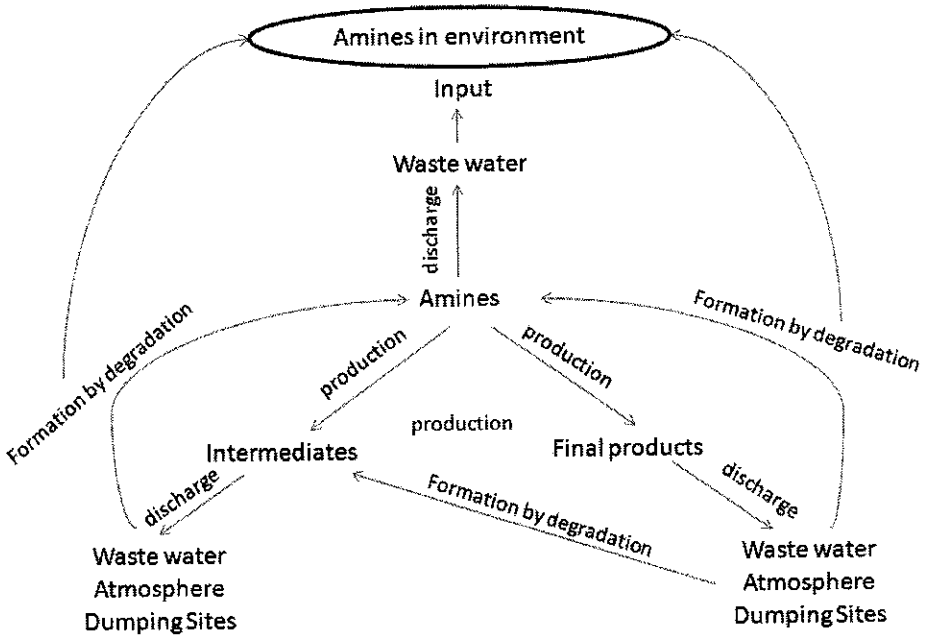

FIGURE 2-2: Emission Sources of Amine into the Water Cycle

2.6 Membrane Process

Membrane is a thin layer of material which serves as a selective barrier between two phases and remains impermeable to specific particles, molecules, or substances when exposed to the action of a driving force. Some components are allowed passage by the membrane into a permeate stream, whereas others are retained by it and accumulate in the retentate stream. (Zydney et al., 1996). The basis advantage of membrane is the solute separation is affected from the solvent without a phase change as compared to conventional methods of evaporation and crystallization (Davis and Berner, 2004).

2.7 Membrane Classes

Membrane classes can be broadly classified on the basis of pressure driving force which works by the application of hydrostatic pressure namely;

- Reverse osmosis (RO),
- Microfiltration (MF)
- Ultrafiltration (UF)
- Nanofiltration (NF)

Membrane filtration technology has developed both in the way membranes are packaged and in the type of material used. The result is a wide range of module configurations and membrane geometries, which are suited to a variety of applications. Polymeric membranes account for biggest proportion of installed membranes currently in use. Several different polymers are used to suit the molecular weight cut off required, or achieve the desired resistance to fouling or performance when contacted with a specific process fluid. Common polymers include; polysuphone and polyethersuphone which are used for the full range of UF membranes.

Polyvinylidene fluoride, PVDF is often used for open UF membranes, whilst polyamide is used as the thin film membrane layer in NF and RO membranes. Cellulose acetate, the first polymer widely used for membranes, is still used in some applications where it exhibits superior fouling characteristics, but its use is limited due to its tendency to hydrolyze in alkaline conditions. Membranes can be configured in tubular, spiral, flat sheet or hollow fiber arrangements. In this case, tubular membrane will be used for experiment.

Tubular membranes, which has several advantages prevent blockage from occurring without the need for prefiltration of the must. They can handle viscous liquids with high level of suspended solids and can be chemically or mechanically cleaned in-place. The tubular polymeric membranes are housed in modules of stainless steel or plastic. This turbulent crossflow performance and large bore tubular design, may eliminate the need for some prefiltration steps and should routinely handle high solids levels.

2.7.1 Reverse Osmosis (RO)

Reverse osmosis is a filtration process that use pressure to force a solution through a membrane. RO is a non-thermal process consisting of dewatering by the separation of pure water from liquid solutions to diffuse through a polymeric membrane. The membrane is impervious to large molecules and retains the valuable components in the must. The process can operate at any temperature between 2°C and 80°C, and since there is no change of phase, it is energy-efficient. Liquid flow within the system is tangential to the membrane surface thus inhibiting formation of deposits which would reduce processing capacity. RO is regarded as the most economical desalination process and played crucial roles in obtaining fresh water from nonconventional water resources such as seawater and wastewater. RO membranes have been used widely for water treatment such as ultrapure water makeup, pure boiler water makeup in industrial fields, brackish water desalination wastewater treatment and reuse in industrial. Loeb invented cellulose-acetate-based RO membrane and had been developed by U.S and Japanese companies to spiral-wound membranes elements using the cellulose acetate asymmetric flat-sheet membranes (Li et al., 2008).

2.7.2 Microfiltration (MF) and Ultrafiltration (UF)

Microfiltration, MF and ultrafiltration, UF membranes having range from several nanometers to micrometer in screening pore size. Since filtration separation by membrane uses pressure different as the driving force for separation and is not accompanied by phase change, it has the following advantages:

1. Separation measure is a low-energy consumption type
2. Target to be separated is scarcely denatured or decomposed due to separation under mild conditions

The performance of MF/UF is dependent on pore size and economic efficiency. Smaller size is not necessarily better because it may be necessary to concentrate a valuable and also necessary to purify by permeating a valuable. Second factor, economic must be adequate to allow for the variations in pore size needed. The efficiency can be represented by:

$$\text{Economical efficiency} = \text{Water permeability} \times \text{Life/Price} \qquad (2.2)$$

The industrial use of MF/UF has been progressed widely such as a clarification procedure in water supply and sewage field, where sand filtration had been conventionally used.

2.7.3 Nanofiltration (NF)

Nanofiltration, NF is a pressure driven separation process that takes place on a layer form by organic membrane. The specific features of NF membranes are mainly the combinations of very high rejections for multivalent ions (>99%) with low moderate rejections for monovalent ions (0-70%), and the high rejection (>90%) for organic compounds with a molecular weight above the molecular weight of the membrane, which is usually in the range of 150-300 (Li et al., 2008).

The traditional material used for NF membranes are organic polymers. Phase inversion membranes are homogeneous and asymmetric and often made of cellulose acetate or poly(ether)sulfone. NF membranes contained functional groups that can be charged, depending on the pH of the solution in contact with the membrane (Li et al., 2008). Application of NF is mostly described in softening of surface water and groundwater. As a softening process, NF is in competition with traditional water-softening processes such as inorganic and organic ion exchange systems, as well as processes such as cold and lime softening and pellet softening (Li et al., 2008).

Benefits of Nanofiltration

- Desalting and concentration within the same unit operation
- Improved product quality, including increased dye strength
- Higher yields - for products which do not require isolation by salting out and filtration by filter presses
- Savings in raw materials, or recovery of products from waste
- Increased dryer capacity and/or education in energy consumption by preconcentration.

Factors that influence the performance of membrane process are pore size of material, driving force and type of membrane. TABLE 2-3 gives an overview and classification of membrane separation processes;

TABLE 2-2: Size of Materials Retained, Driving Force and Type of Membrane (Perry, Green, 1997)

Process	Size of Material Retained	Driving Force	Type of Membrane
Reverse Osmosis	<1 nm molecules	Pressure difference (10-100 bar)	Nonporous
Microfiltration	0.1 – 10 μm microparticles	Pressure difference (0.5-2 bar)	Porous
Ultrafiltration	1-100 nm macromolecules	Pressure difference (1-10 bar)	Microporous
Nanofiltration	0.5-5nm molecules	Pressure difference (10-70 bar)	Microporous

Membrane performance is often measured by the ability of the membrane to prevent, regulate or facilitate permeation. The rate of permeation and the mechanism of transport depend upon the magnitude of the driving force, the size of the permeating molecule relative to the size of the available permanent or dynamic transport corridor and the chemical nature (dispersive, polar, of both the permeate and the polymeric membrane material (Lloyd, 1985).

2.8 Types of Flow

In membrane separation, there are two types of flow, which are crossflow and dead-end flow.

2.8.1 Dead-End Flow

Dead-end flow is a filtration technique in which all the fluid passes through the membrane and all particles larger than the pore size of the membrane. The water that enters the membrane surface is pressed through the membrane. Some solids and components will stay behind on the membrane while water flows through. This depends on the pore size of the membrane. Consequentially, the water will experience a greater resistance to passing through the membrane. When feed water pressure is continual, this will result in a decreasing flux. After a certain amount of time the flux has decreased to such an extent, that the membrane will need cleaning.

Dead-end management is applied because the energy loss is less than when one applies a crossflow filtration. This is because all energy enters the water that actually passed the membrane. The pressure that is needed to press water through a membrane is called Trans Membrane Pressure, TMP. The TMP is defined as the pressure gradient of the

membrane, or the average feed pressure minus the permeate pressure. The feed pressure is often measured at the initial point of a membrane module. However, this pressure does not equal the average feed pressure, because the flow through a membrane will cause hydraulic pressure losses.

During cleaning of a membrane, components are removed hydraulically, chemically or physically. When the cleaning process is performed, a module is temporarily out of order. As a result, dead-end management is a discontinuous process. The length of time that a module performs filtration is called filtration time and the length of time that a module is cleaned is called cleaning time. In practice one always tries to make filtration time last as long as possible, and apply the lowest possible cleaning time. When a membrane is cleaned with permeate, it does not have a continuous production of water. This results in a lower production. The factor that indicates the amount of production is called recovery.

2.8.2 Crossflow Flow

Crossflow is a type of filtration when feed flow travels tangentially across the surface of the filter and avoid solids being trapped on and in the filter. When crossflow filtration takes place, feed water is recycled. During recirculation, the feed water flow is parallel to the membrane. Only a small part of the feed water is used for permeate production, the largest part will leave the module. Consequentially, crossflow filtration has a high energy cost. After all, the entire feed water flow needs to be brought under pressure. The water speed of the feed water flow parallel to the membrane is relatively high. The purpose of this flow is the control of the thickness of the cake.

Consequentially to the flow speed of the water, flowing forces are high, which enables the suspended solids to be carried away in the water flow. Crossflow management can achieve stable fluxes. Still, the cleaning of crossflow installations needs to be applied from time to time. Cleaning is performed by means of backward flushing or chemical cleaning. The crossflow system is applied for RO, NF, UF and MF, depending on the pore size of the membrane. TABLE 2-2 below summarizes the advantages and disadvantages of dead-end flow and crossflow and FIGURE2-3 shows the illustration of both flows..

TABLE 2-3: Advantages and Disadvantages of Dead-end and Cross-flow Filtration

	Advantages	Disadvantages
Dead-end flow	• High collection rate (almost 100%) • Miniaturization possible • Low cost • Backwashing and chemical cleaning not required	• Filters must be replaced often • Can not be used if large amounts of insoluble materials are present
Cross-flow flow	• Low filter maintenance frequency • Can be used even if large amounts of insoluble materials are included • Can be used for viscous liquids as well • Can be reused with backwashing and chemical cleaning	• Low collection rate, due to separation into filtered water and concentrated water • Treatment of concentrated water is required • Unit is large and complicated • Relatively high cost

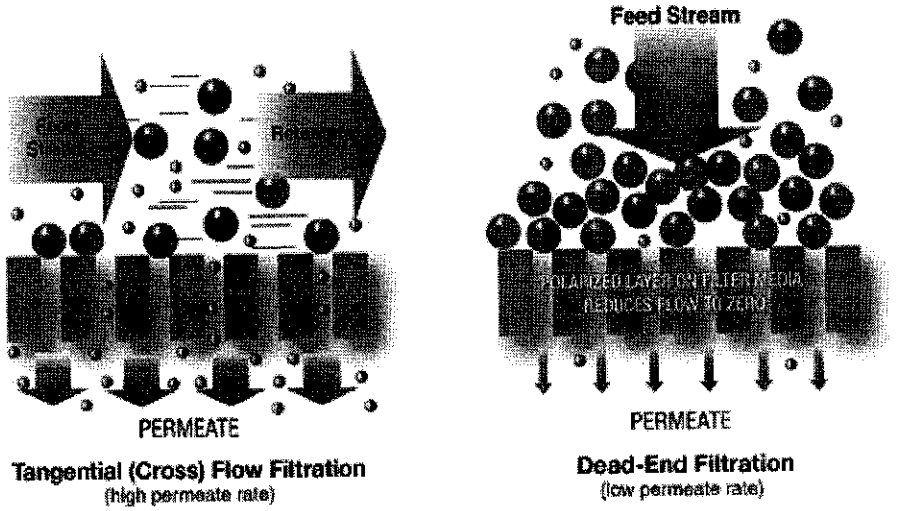

FIGURE 2-3: Cross-flow and Dead-end Flow

Cross-flow membrane technology:

- Reduces pollutants and contaminants
- Meets local water discharge legislation
- Increases efficiency and effectiveness of biological treatment systems
- Provides cost effective of treating leachate and treats variety of leachate types
- Designed to meet specific site demands, for example fluctuations in volumes and composition

2.9 Factors Affecting Membrane Performance

2.9.1 Concentration Polarization (CP)

The concentration polarization reduces the permeating component's concentration difference across the membrane, thereby lowering its flux and the membrane selectivity. CP can significantly affect membrane performance in RO but it is usually controlled, unlike in UF and electro dialysis process whereby the membrane performance is seriously affected (Baker, 2000). The size of solute diffusion coefficient explains why CP is a greater factor in UF than in RO. In RO, the solutes are dissolved salts whereas in UF, the solutes are colloids and macromolecules. The diffusion coefficients of these high-molecular-weight components are about 100 times smaller than those salts.

2.9.2 Pressure

Operating pressure affects both water flux and salt rejection of membrane. Water flux across membrane has direct relationship increasing operating pressure. Increased operating pressure also results in increased salt rejection but the relationship is less direct than water flux. There is always some salt passage is increasingly overcome as water is pushed through the membrane at a faster rate than salt can be transported. However, there is an upper limit to the amount of salt that can be excluded increasing operating pressure. Above a certain pressure level, salt rejection no longer increases and some salt flow remains coupled with water flowing through the membrane (Baker, 2000).

2.9.3 Feed Concentration

Osmotic pressure is a function of the type and concentration of salts or organics contained in water. As a feed concentration increases, so does the osmotic pressure. If the operating pressure remains constant, higher feed concentration results in lower membrane flux. The increasing osmotic pressure offsets the feedwater driving pressure and as the water flux declines, salt rejection also decrease.

2.9.4 Temperature

As water temperature increases, water flux increase almost linearly due primarily to the higher diffusion rate of water through the membrane. Increased operating temperature also results in lower salt rejection or higher salt passage. This is due to a higher diffusion rate for salt through the membrane.

2.9.5 Recovery

If percentage recovery is increased, and the operating pressure remains constant, the salt in the residual feed become more concentrated and the natural osmotic pressure will increase until it is as high as the applied feed pressure. This can negate the driving effect of operating pressure, slowing or halting the reverse osmosis process and causing permeate flux and salt rejection to decrease. The maximum percent recovery possible in any membrane system usually depends on the concentration of salts present in the feedwater and their tendency to precipitate on the membrane surface as mineral scale.

2.9.7 pH

Membrane salt rejection performance also depends on pH, which may also affect the water flux. Rejection of weak acids and bases especially in UF is highly dependant. This change in rejection behavior is related to the change in configuration of the acid. When the acid or base is in ionized form, the rejection will be high but in nonionized form the rejection will be low (Matsuura, Sourirajan, 1972). In ionized form, the negatively charged carboxyl groups along the polymer backbone repel one another, the polymer coil is then very much extended and relatively inflexible. In this form, the molecule can not readily permeate the small pores in the membrane. At low pH, the carboxyl group along the acid polymer is protonated. The resulting neutral molecule is much more flexible and therefore can pass through the membrane. The pH tolerance of various types of membrane can vary widely. pH of most naturally occurring water through a RO membrane is within 7 to 9.5. Thin film composite membranes are typically stable over a broader pH range than cellulose acetate, CA and therefore, offer greater operating latitude.

CHAPTER 3

3.0 MATERIAL AND METHODS

3.1 Material

The experimental work has used the following materials;

a. Triethanolamine, TEA

TABLE 3-1 shows the physical and chemical properties of TEA. Detailed properties were shown in APPENDIX A.

TABLE 3-1: Properties of TEA

Properties	Triethanolamine, TEA
Chemical	$(C_2H_5O)N_3$
Molecular Weight, g/mol	149.19
Purity	99%
Appearance	Colorless to pale yellow
Odor	Ammonia
Boiling Point, °C	335
pH	10.5 (15g/L H_2O)

b. Membrane

The Membrane Test Unit has 4 containers which can put 4 membranes at the same time. TABLE 3-2 shows the properties of membrane used in the project. Detailed information for membrane was shown in APPENDIX B.

TABLE 3-2: Membrane Information

Membrane Type	Membrane Material	Max pH range	Max pressure, bar	Max temperature, °C	Apparent Retention Character[1]	Pore Size (nm)
AFC99	Polyamide Film	1.5-12	64[2]	80	99% NaCl	<0.6
AFC40	Polyamide Film	1.5-9.5	60	60	60% $CaCl_2$	0.6-5
CA202	Cellulose Acetate	2-7.25	25	30	2000 MW	5-50

[1] Retention character depends on several parameters, including nature of the test solution

[2] Maximum pressure limited by module

3.2 Methods

The experiment of amine separation by using membrane was characterized into several parts where steps were indentified first in order to obtain accurate data and work in appropriate manner. Before starting the experiment, the parameters that will affected the separation process were identified, such as operating pressure, cross-flow velocity, types of membrane and chemical, and feed concentration of solution.

The experiment started with the following procedures;

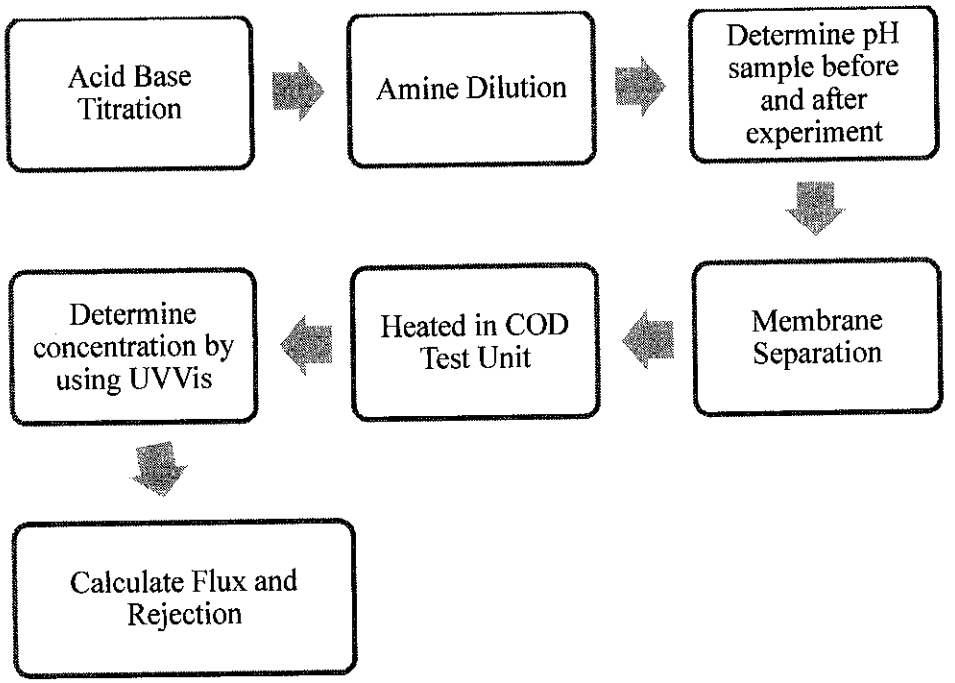

FIGURE 3-1: Flow Diagram of Experimental Procedure

3.3 Parameter

a) Pressure

The pressure used in this experiment was proposed up to 20bar. This is because membrane CA202, Cellulose Acetate has the maximum pressure at 25bar. If higher pressure was to be put on the membrane, it will break down the membrane. Although, the Membrane Test Unit has the capability to operate up to 60bar. But after some run by using tap water, the equipment leaked and can only be operated up to 25bar. For safety measure, lower pressure was used, which was at 20bar. Every parameter of operating pressure was repeated at same constants. The following operating pressure was selected for the experiment; 4, 8 12, 16 and 20 bar.

b) Cross-flow Velocity

The cross-flow velocity in membrane separation affects the process. Higher cross-flow velocity tends to result in higher flux where the flow will push down the molecule to pass the membrane pores. In this case, the values used were 2, 4, 6 and 8 LPM (liter per minute).

c) Feed Concentration

Wastewater from sweetening process was characterized by up to 52,000 ppm COD, and in certain case can reach of 350,000 ppm COD. Based on this information, the highest range of feed concentration parameter is 52,000ppm. By dividing the concentration value by half, it will lead to lower concentrations that were used in experiment. The following feed concentration was selected; 6500, 13 000, 26 000 and 52 000ppm.

3.4 Equipment/Apparatus

The separation study was done by membrane by using Membrane Test Unit. After run the experiment, samples were taken for result analysis. COD Test Unit was used for the analysis, which is to determine the COD content in sample, pH meter to determine the pH of solution before and after the experiment and UVVIs, to determine the concentration of sample. TABLE 3-1 below summarizes the equipment used and apparatus for the preparation of amine solution.

TABLE 3-3: The Equipments and Function

No	Equipment/Others	Function
1	Membrane Test Unit	To separate the solutions
2	COD Test Unit	To determine COD content in sample
3	pH meter	To determine sample pH
4	UVVis	To determine concentration of sample
5	Beaker	To place chemicals/solution
6	Pipette	To transfer small amount of chemical
7	Burette	To measure and separate known chemical in certain amount
8	Retort Stand and Clamp	To hold apparatus in laboratory
9	Erlenmeyer Flask	For titration of acid base
10	Pail	To mix chemicals and distilled water

For acid base titration, hydrochloric acid and phenolphthalein were used as well as distilled water for amine dilution. Every sample was analyzed by mixed it with 2ml of COD reagent. TABLE 3-4 below summarizes the chemicals and the recommended quantity.

TABLE 3-4: The List of Chemicals

No	Chemicals	Quantity
1	Triethanolamine, TEA	100ml
2	Hydrochloric Acid, HCL	20 ml
3	Distilled water	1000 L
4	COD Reagent	300 ml
5	Phenolphthalein	10ml

3.5 Procedure

The procedure of the experiment started at acid base titration process. For acid base titration, amine was initially in base condition. To avoid working in hazardous condition because amine has high base and membrane capability, amine was neutralized by using hydrochloric acid, HCL and phenolphthalein as pH indicator.

Before run the experiment, the chemicals used need to be extra handling because they are hazardous and corrosive. Amine can cause burns and may lead to irritation or dermatitis. High volatility can cause serious damage to eyes. Below shown the procedure of acid base titration;

Titration of acid base

1. An amount of amine was poured (calculated from correlation below) into Erlenmeyer flask.

$$Concentration, \frac{g}{L} = \frac{Volume\ amine, L\ x\ Density\ Amine, g/L}{Volume\ Water, L} \quad (3.1)$$

2. Pure amine was diluted with 300mL of distilled water.
3. Phenolphthalein was added to the amine, where the solution will become pink in color.

4. HCL was added to burette. The burette was set over the flask so the titrant can be added in a controlled manner to the amine. The initial point of HCL was marked.
5. pH for amine was checked by using pH meter.
6. Slowly, HCL was added to the solution until the indicator changes color to colorless.
7. The final point of HCL was measured and determined the volume of HCL used to neutralize the amine.

After titration process, amine was diluted into 20L of distilled water. The 20L of amine solution will be using continuously for about 10 minutes of each run. Below shown the procedure of amine dilution;

Preparation of amine solution

1. The amine-after-titration was prepared in large container.
2. 20L of distilled water was added into the container.
3. The solution was properly mixed by using long stick.
4. Before start the experiment, pH of solution was measured by using pH meter.

The solution was poured into feed tank of Membrane Test Unit. The parameters on this experiment were feed concentration, operating pressure and cross-flow velocity. The experiment started with 6500 ppm , 4 bar and 2 LPM. Below shown the procedure of using the equipment;

Experimental Procedure for using Membrane Test Unit

1. TEA solution was prepared as explained in above section. All valves were closed except V2, V5, V8, V10, V12 and V14.
2. The feed tank was filled up with the solution prepared in Step 1. The feed shall always be maintained at room temperature.
3. The maximum working pressure was set up at 20 bars.

Note: For working pressure setting, valve, V5 was closed. A proper wrench was used to turn the adjusting screw at the pressure regulator by turning clockwise to increase and counter-clockwise to reduce the pressure.

4. The plunger pump, P1 was started. The membrane maximum inlet pressure was set to 4 bars by adjusting the retentate control valve (V15).
5. The system was allowed to run for 10 minutes. The collecting sample started from permeates sampling port. The weight of permeate was recorded every 1 minute for 10 minutes.

   Note: The sample was collected by open valves V7, V9, V11 and V13, simultaneously closed valves V8, V10, V12 and V14.
6. The plunger pump, P2 was stopped and valve, V2 was closed.
7. Data of experiment was collected.

The procedure was repeated for different feed concentration, operating pressure and cross-flow velocity. Sample of each membrane was taken to be tested by using COD Test Unit, pH meter and UVVis. Sample was mixed with 2ml of COD reagent before being tested by the test unit as follows;

Preparing the COD Vial

1. COD vial was opened.
2. A blank sample was prepared by using distilled water.
3. The cap was replaced tightly.
4. The vial was gently shaked to properly mix the contents.

Procedure of using COD Test Unit

1. The start up of the test unit was performed as shown in APPENDIX D.
2. The instrument was switched on.
3. The required temperature program was selected at 150°C for two hours.
4. The test vials were prepared as described in the above section.
5. The instrument was heated to the set temperature. Two beeps indicate that the required temperature has been reached.
6. The vials were placed in the appropriate heating block and the protective lid was closed.

7. The program was started using the left key.
8. The time automatically counts down to zero (0). The actual temperature and the remaining time were visible on the display.

Clock Symbol	Description
	The remaining time counts down to zero (0).

9. The instrument will beep three times to indicate the temperature program is complete. It will turn off the heater and cool. During operation, the display will indicate the status of the thermometer.

Thermometer Symbol	Description
	Heating block is heating.
	Heating block has reached the set temperature.
	Heating block is cooling.

3.6 Result Analysis

The data obtained were analyzed following the diagram in FIGURE 3-1.

For 10minutes, the volume of permeate for each membrane was taken in time interval. The data will be analyzed for flux and rejection.

TABLE 3-5: Table of Data Collected

Amine = TEA, Concentration = 6500ppm, P = 2bar

	Membrane Type	Time, s									
		60	120	180	240	300	360	420	480	540	600
Volume, ml	AFC99										
	AFC40										
	CA202										

pH of sample was checked to determine whether the amine pH change before and after the separation. pH is one of the factor of membrane separation. In this case, the pH used was neutral for safety measure. Then, the COD content was checked by UVVIs to determine the level COD and amine separated by each membrane.

After separation, the amine-solution concentration was changed. Equipment UVVIs was used to determine the after-concentration of amine solution. The data were analyzed by calculating the flux, J by below correlation;

$$Flux, J \left(\frac{L}{m^2.\,\mathrm{h}}\right) = \frac{Volume}{Area\,.Time}$$

$$= Weight, g \; x \, Density, {}^{L}/_{g} \; x \; {}^{1}/_{(Area, m^2 \; x \; Time, s)} \tag{3.2}$$

The area for membrane is;

$$\text{Area, } m^2 \quad = 2\pi rL \tag{3.3}$$
$$= 2\pi \, (0.00625\text{m}) \, \text{x} \, (1.2\text{m})$$
$$= 0.0471 \; m^2$$

Graph of flux versus operating pressure at feed concentration, cross-flow velocity and three types of membrane were plotted. Rejection of separation was calculated by using below correlation;

$$Rejection, R \; = \; \left(1 - \frac{C_p}{C_b}\right) x \, 100\% \tag{3.4}$$

Rejection is ratio of concentration of after-separation, C_b with initial concentration, C_p of solution. Rejection determines how much molecules of amine has not passed the membrane pores. Higher rejection shows good separation where more chemical is separated.

3.7 Safety/Precaution

For spill control, during and after the experiment, safety and precaution were taken care in detailed manner because amine is very corrosive and very harmful to human. Amine will become slippery when spilt. Protective equipments were worn to prevent skin and eye contact and breathing in vapors. Wind or increase ventilation was worked up. To avoid accidents, any spilt was cleaned up immediately. Spill was absorbed with inert material absorbent such as soil, sand or earth. Containers or drums for disposal were properly collected and sealed. Amine residue can be neutralized with dilute acetic acid. Area down was washed with excess water.

3.8 Membrane Test Unit

The membrane Test Unit was specially designed to carry out the membrane processes that are widely used in biotechnology and process industries such as RO, UF and NF. The process diagram was illustrated in APPENDIX C.

UF membranes are usually specified in terms in terms of their molecular-weight-cut-off, MWCO, whereas the NF and RO membranes are specified in terms of their percentage rejection of salts. Polymeric membranes are widely used and supplied in the form of modules that give membrane areas in the range of 1-20m^2. The membranes that are supplied with the Membrane Test Unit are classified as tubular type, which is widely used and have turbulent flow conditions. The system is in a cross flow configuration where the feed solution is pumped parallel to the membrane at a velocity in the range of 1-8ms^{-1} with a pressure difference of 0.1-0.5MPa across the membrane. Liquid permeates through the membrane and feed emerges in more concentrated form on the exit module.

3.8.1 Membrane and Membrane Housing

Single-tube Tester was designed for the economical, quick, initial evaluation of membrane types and processes for separation and concentration at laboratory scale prior to more details test work. It may be fitted with samples of wide range of tubular RO, NF and UF membranes. Simply constructed in 316-stainless-steel, the module has termination points allowing easy connection by flexible or welded couplings to existing equipment. The open channel, highly turbulent flow design allows a wide variety of processes fluid to be concentrated. It also allows simple clean-in-place techniques to be entirely effective. The membrane information can be obtained in APPENDIX B. The CA 202 Cellulose Acetate membrane is rated with apparent retention character of 2000 MWCO. In addition, the Membrane 2, Polyamide Film has 60% $CaCl_2$ and the Membrane 2, Polyamide Acetate is rated with 99% NaCl rejection.

CHAPTER 4

4.0 RESULT AND DISCUSSION

In this chapter, the writer will discuss about the result and discussion based on data obtained from experimental work. Below is the result for membrane process by using water and Triethanolamine, TEA.

4.1 Water Permeability

This was the study of water permeability. Detailed data on water permeability was shown in APPENDIX E.

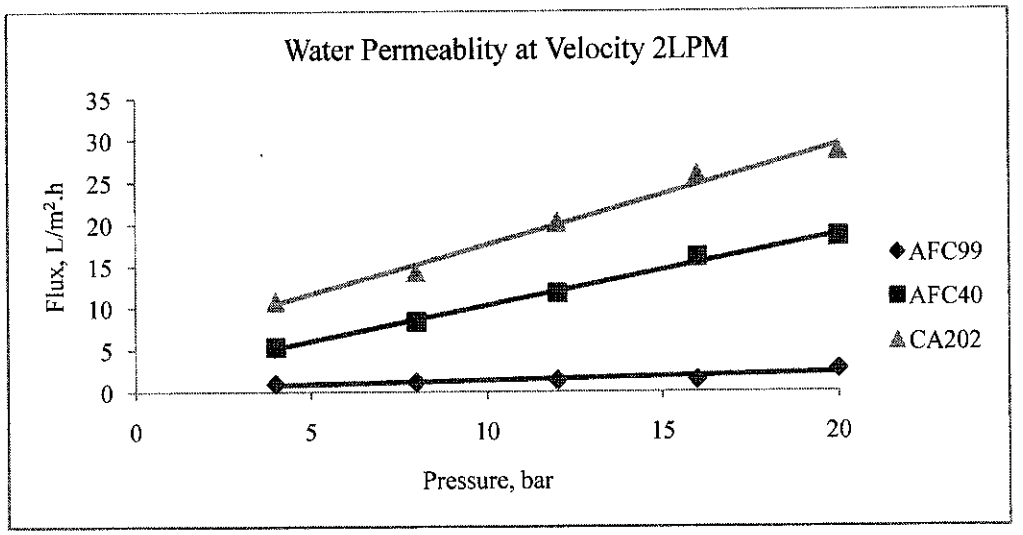

FIGURE 4-1: Water Permeability Graph at Constant Velocity 2 LPM

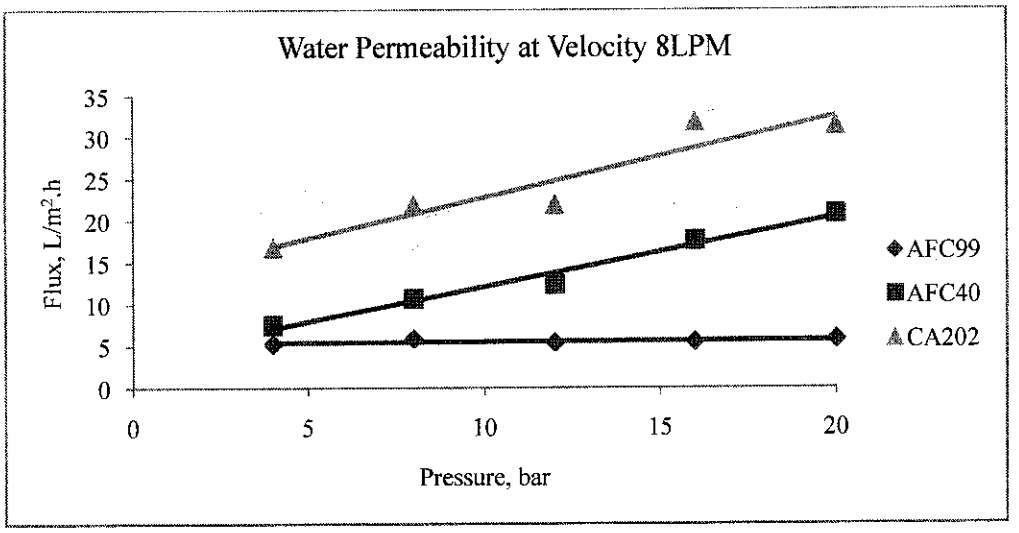

FIGURE 4-2: Water Permeability Graph at Constant Velocity 8 LPM

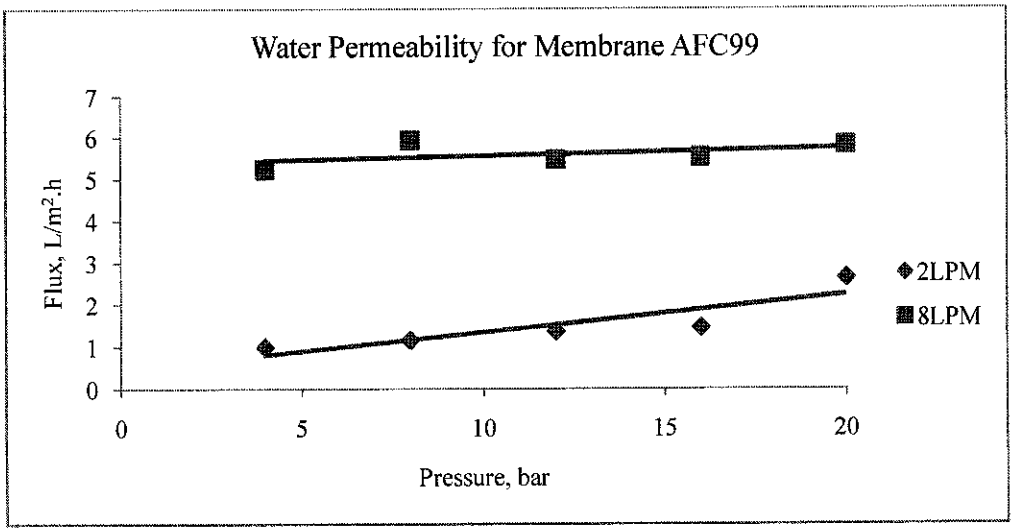

FIGURE 4-3: Water Permeability Graph for Membrane AFC99

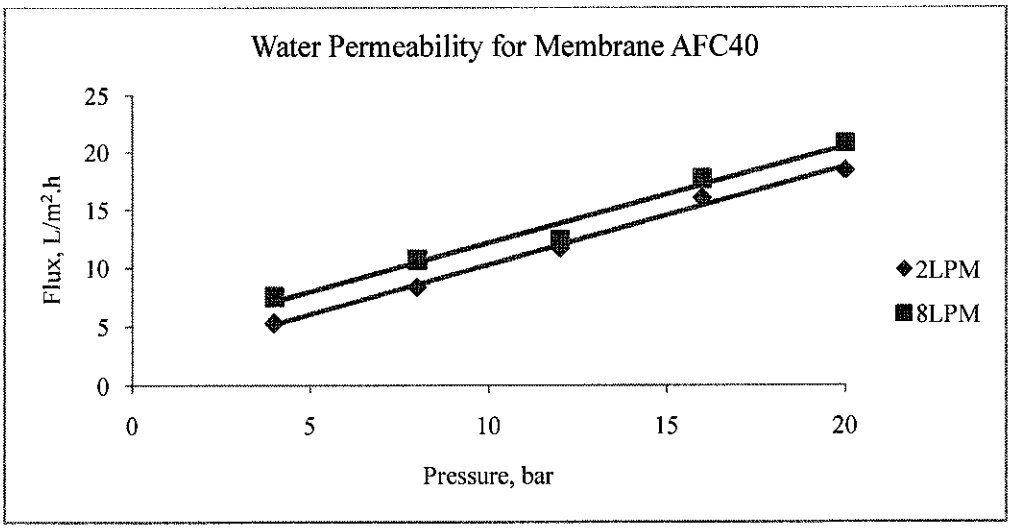

FIGURE 4-4: Water Permeability Graph for Membrane AFC40

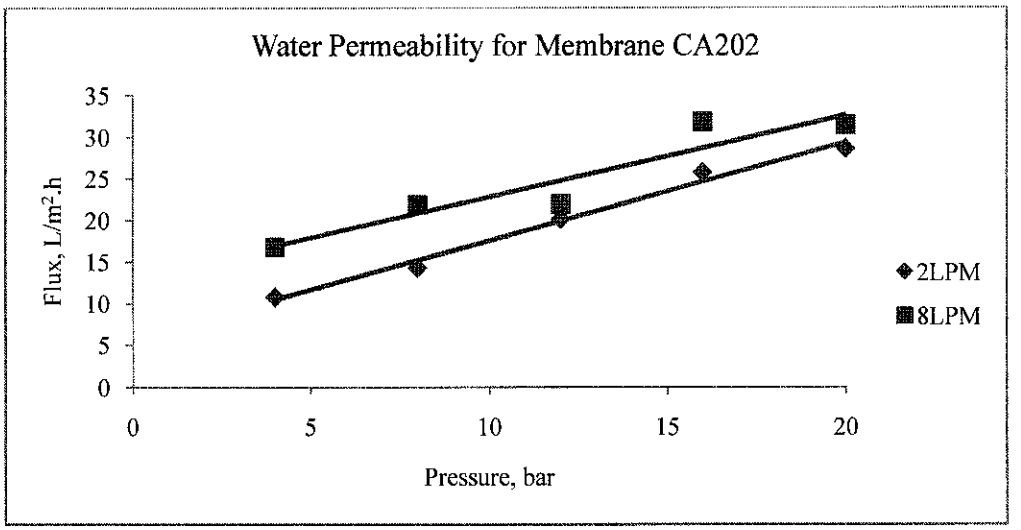

FIGURE 4-5: Water Permeability Graph for Membrane CA202

Permeate flux is important parameter in the design and economical feasibility analysis of the membrane separation process. Flux is affected by several factors such as operating pressure, cross-flow velocity or feed composition (Koyuncu *et al*, 2001; Chen *et al*, 1997).

FIGURE 4-1 shows the effect of operating pressure of membranes. Water flux across the membrane increases in direct relationship to increase in operating pressure. Increased operating pressure also results in increased salt rejection but the relationship is less direct than water flux. Cross-flow velocity plays important role in increasing flux. For membrane AFC99, as operating pressure increases, flux increases. Similarly,

flux was increased as cross-flow velocity increased. Same pattern goes for membrane AFC40 and CA202 in FIGURE 4-2 and 4-3, where flux increases when operating pressure increases and cross-flow velocity increases. But the flux value is much greater then AFC99 and highest flux is for CA202, followed by AFC40. FIGURE 4-4 and 4-5 show comparison between three membranes at 2LPM and 8LPM. The figures clearly show that membrane CA202 has highest flux, followed by AFC40 and AFC99 at both velocities.

Above a certain pressure level, salt rejection no longer increases and some salt flow remains coupled with water flowing through the membrane (Baker, 2000). But for this case, the operating pressure is up to 20bar and can not go further because of equipment capability. The experiment by using amine, TEA is used by using the same parameter with addition of feed concentration variables. Section 4.2 shows the results obtained in term of permeate flux by comparing highest and lowest value of parameter for operating pressure, cross-flow velocity and feed concentration.

4.2 TEA Separation in Term of Flux

For amine separation, detailed data on all parameters are shown in APPENDIX F.

a) TEA Concentration 6500ppm

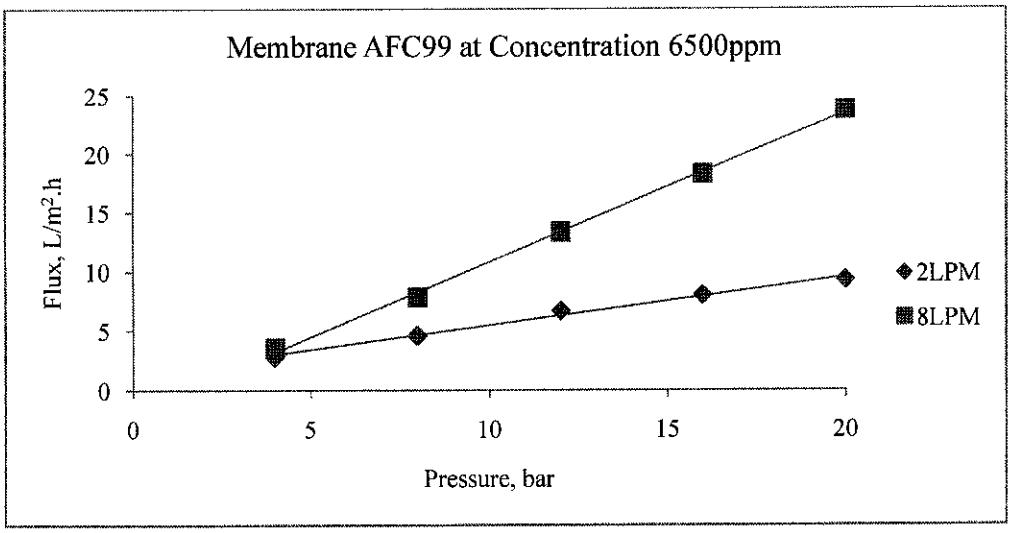

FIGURE 4-6: Flux vs. Pressure at Constant Concentration 6500ppm for Membrane AFC99

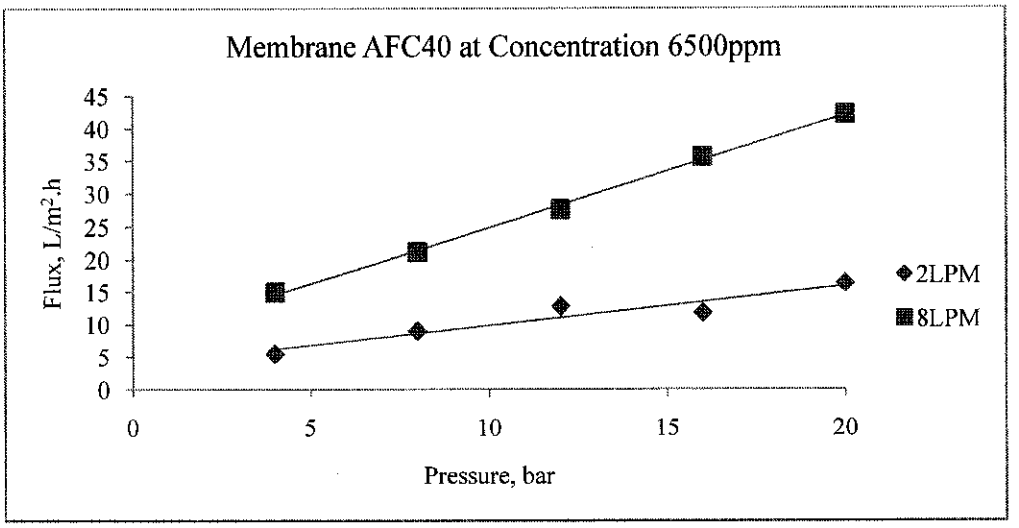

FIGURE 4-7: Flux vs. Pressure at Constant Concentration 6500ppm for Membrane AFC40

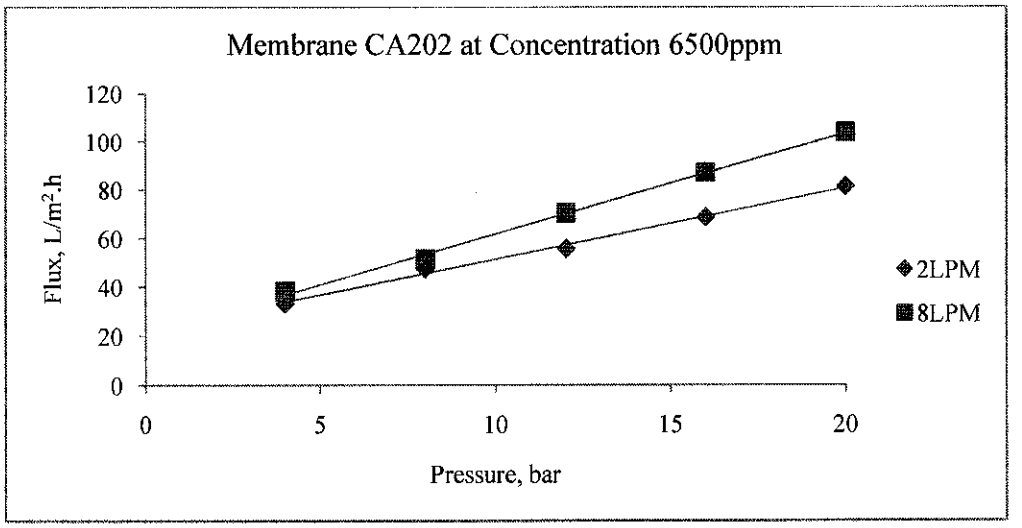

FIGURE 4-8: Flux vs. Pressure at Constant Concentration 6500ppm for Membrane CA202

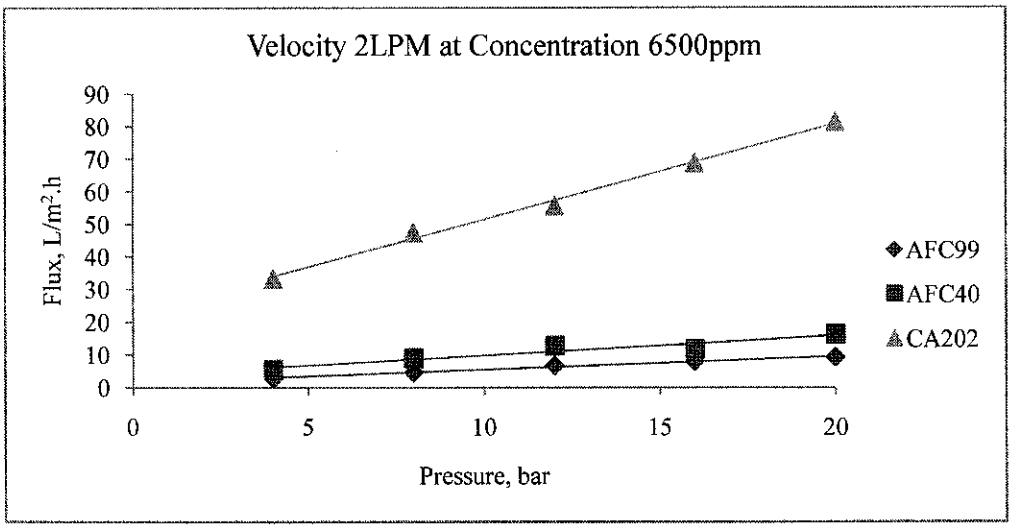

FIGURE 4-9: Flux vs. Pressure at Constant Concentration 6500ppm with Velocity 2LPM

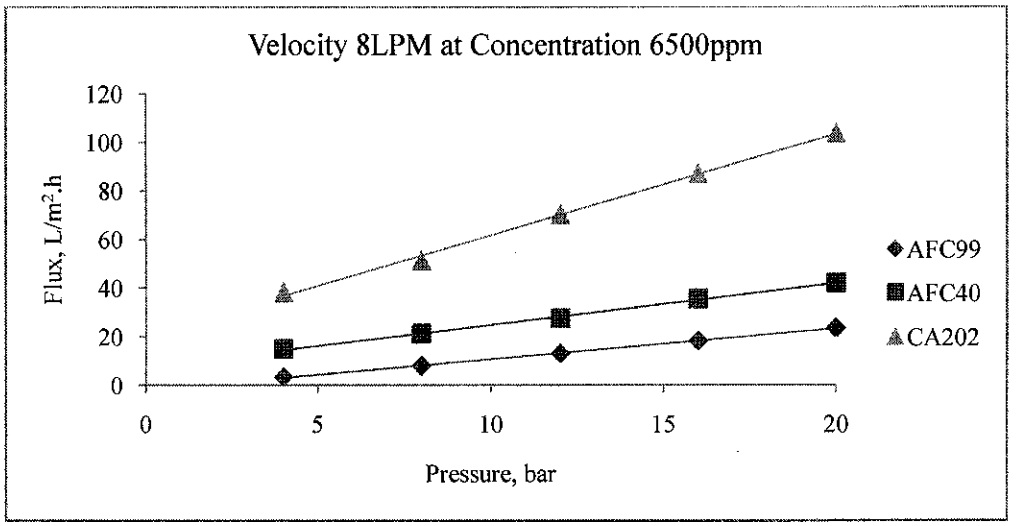

FIGURE 4-10: Flux vs. Pressure at Constant Concentration 6500ppm with Velocity 8LPM

b) Concentration 52 000ppm

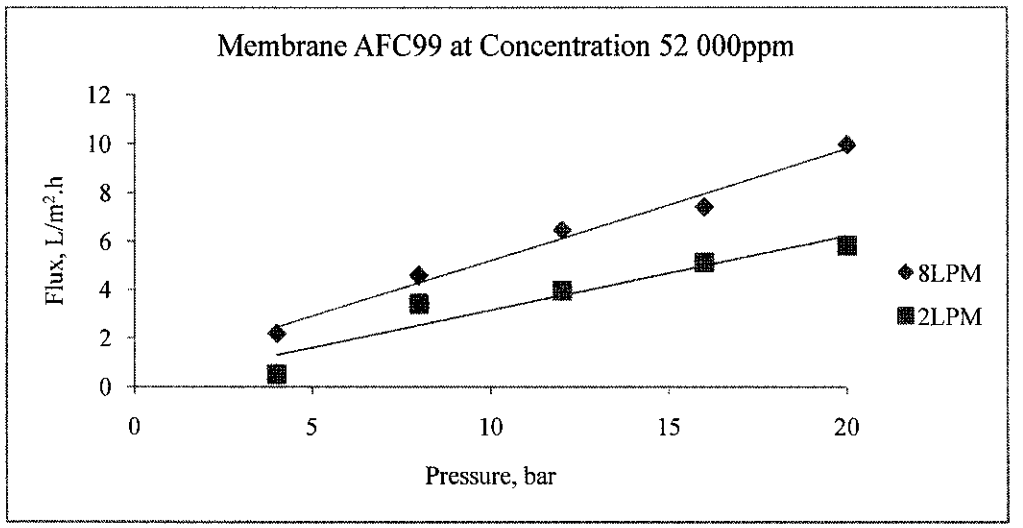

FIGURE 4-11: Flux vs. Pressure at Constant Concentration 52 000ppm for Membrane AFC99

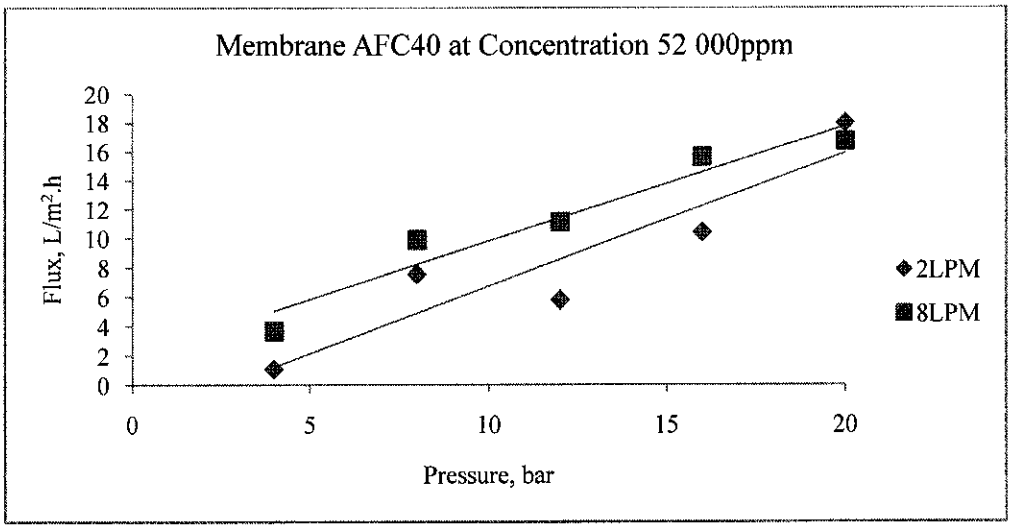

FIGURE 4-12: Flux vs. Pressure at Constant Concentration 52 000ppm for Membrane AFC40

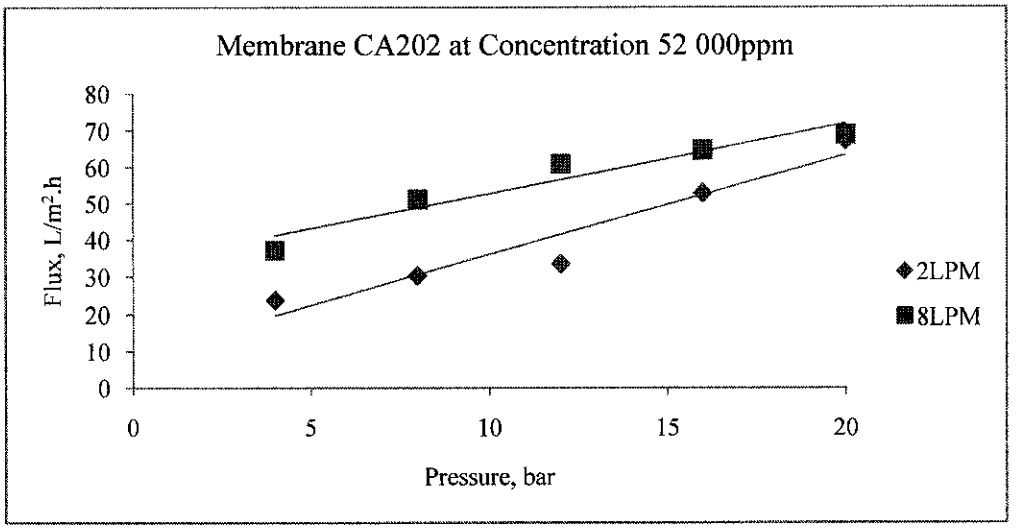

FIGURE 4-13: Flux vs. Pressure at Constant Concentration 52 000ppm for Membrane CA202

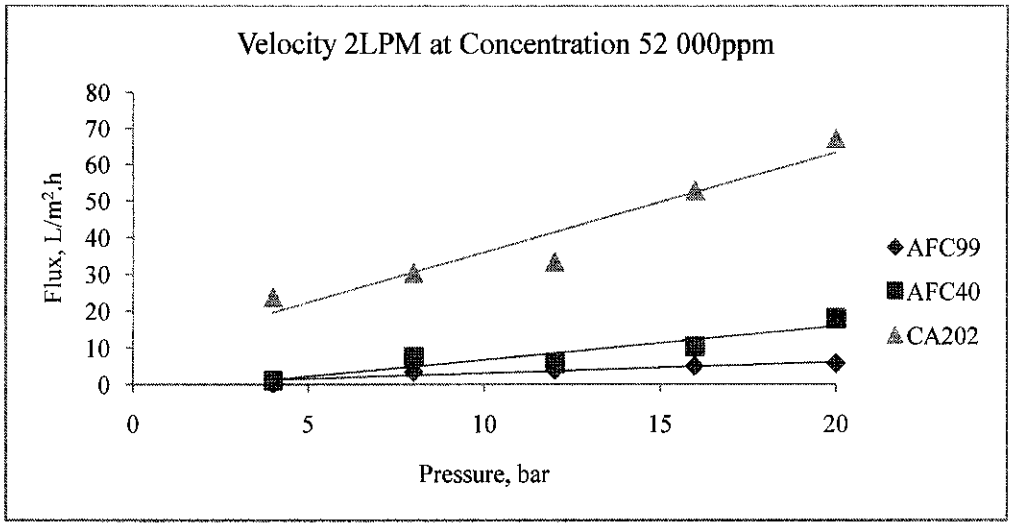

FIGURE 4-14: Flux vs. Pressure at Constant Concentration 52 000ppm and Velocity 2LPM

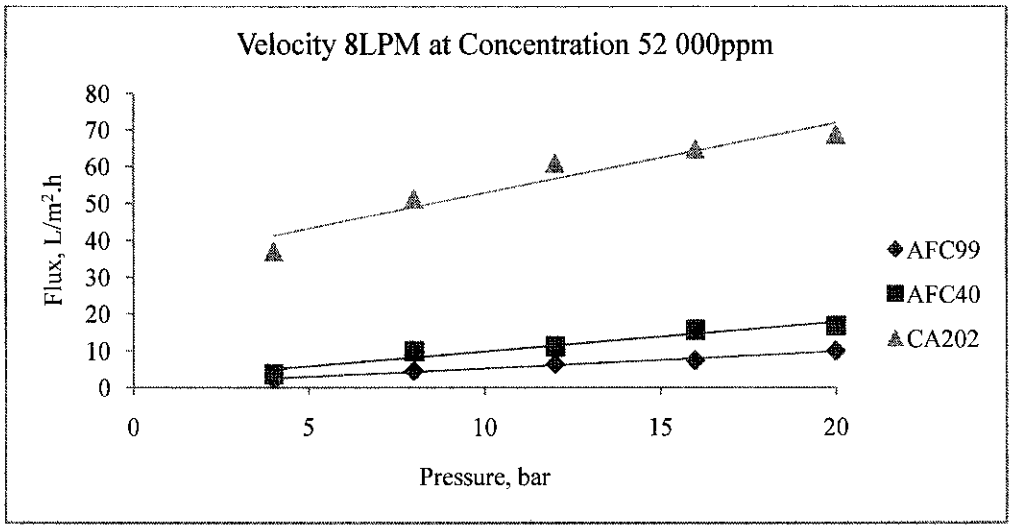

FIGURE 4-15: Flux vs. Pressure at Constant Concentration 52000ppm and Velocity 8LPM

Effect of Cross-flow Velocity

Permeate flux is important parameter in the design and economical feasibility analysis of the membrane separation process. Flux is affected by several factors such as operating pressure, cross-flow velocity or feed composition (Koyuncu *et al*, 2001; Chen *et al*, 1997). Increase in flux can be observed at high cross-flow velocities due to the decrease in concentration polarization (Koyuncu *et al*, 2003). The velocity in membrane

separation affects the process. In FIGURE 4-6, 4-7 and 4-8 shows that higher cross-flow velocity tends to result in higher flux where the flow will push down the molecule to pass the membrane pores at different membranes which were AFC99, AFC40 and CA202. By using cross-flow velocity variable from 2 to 8 LPM, as the cross-flow velocity increased, the flux will be increased. Comparing between the three graphs, flux at membrane CA202 shows highest flux, followed by AFC40 and AFC99. This is because of membrane porosity. The AFC99, which reverse osmosis process, has very tight pore size (<0.6nm). Compare to AFC40, the nanofiltration process, the pore size is in range 0.6-5nm has CA202 has the highest pore size which is 5-50nm. The size determines the flux of the separation process. The effect of the flow rate on the species permeance is detectable when comparing values at the highest and lowest flow rates. This difference is caused by the higher amine transport resistance and stage cut reduction within the membrane, which is caused by reduction in amine residence time at higher flow rates. In this case, the author compared between highest and lowest cross-flow velocity, 2 and 8 LPM respectively as shown in FIGURE 4-9 and 4-10. Obviously, 8LPM gives higher flux than at 2LPM. But, at different concentration, flux decreases as the feed concentration increases. Refer to FIGURE 4-11 to 4-15, it was observed that the flux is decreasing, and this is due to concentration polarization, as will be explained below.

Effects of Concentration Polarization

The increase in flux values caused by an increased in cross-flow velocities is explained by concentration polarization. Refer to FIGURE 4-9 and 4-10, at low amine concentrations, the mass transfer effect caused by concentration polarization is small and easily prevented by increase in cross-flow, thus increasing the flux. Compared with higher concentration, which was at 13000ppm, at FIGURE 4-14 and 4-15, the flux was lower. By using high concentration up to 52000ppm, the flux showed the lowest. This causes the relatively large deviations observed in the flux. The mass transfer effects caused by concentration polarization are much greater due to the increased in concentration at high salt concentrations. In this cases the effects of increasing the cross-flow velocity are small. This is simply due to the fact that small changes in cross-flow velocities are not significant enough to reduce the mass transfer effect due to the increased in concentration polarization of the salt.

Theoretically, concentration polarization exists in all membrane separation processes because of the selective permeability of membrane and tends to have locally extraordinarily high concentration at or near the membrane surface. It has serious adverse effects in membrane separation processes. It leads to a decrease in the driving force for the permeable species, TEA across the membrane. This reduces the overall efficiency of separation and raises the costs of capital and operation. Increasing flow rates will provides a smooth flow on the membrane surface and reduce converge molecules on the membrane area, which do affect the permeability of salt into the permeate stream (Ahmad Fauzi *et al*, 2001). As a result, TEA permeate enrichment increases with increases the retentate flow rate. Generally such concentration polarization is undesirable since it may cause precipitation of the species congregating at the membrane surface leading to pore plugging, reduced flux, and a varying rejection coefficient (Oroskar *et al*, 1991).

Effects of Operating Pressure

Salt permeate enrichment is defined as the permeate salt concentration over feed salt concentration. As shown in FIGURE 4-10 and 4-11, increasing the operating pressure from 4 to 20 bar resulted in the increase of the pressure difference across the membrane as well as the driving force for the separation process. In addition, the increase in species permeability as a function of pressure was caused by the increase in species solubility and diffusivity, with higher values for TEA of the faster permeating species. Hence, more TEA diffused through the membrane and as a result, the permeate purity increases.

4.3 TEA Separation in Term of Rejection

For amine separation, detailed data on all parameters were shown in APPENDIX F.

a) At feed concentration 6500ppm

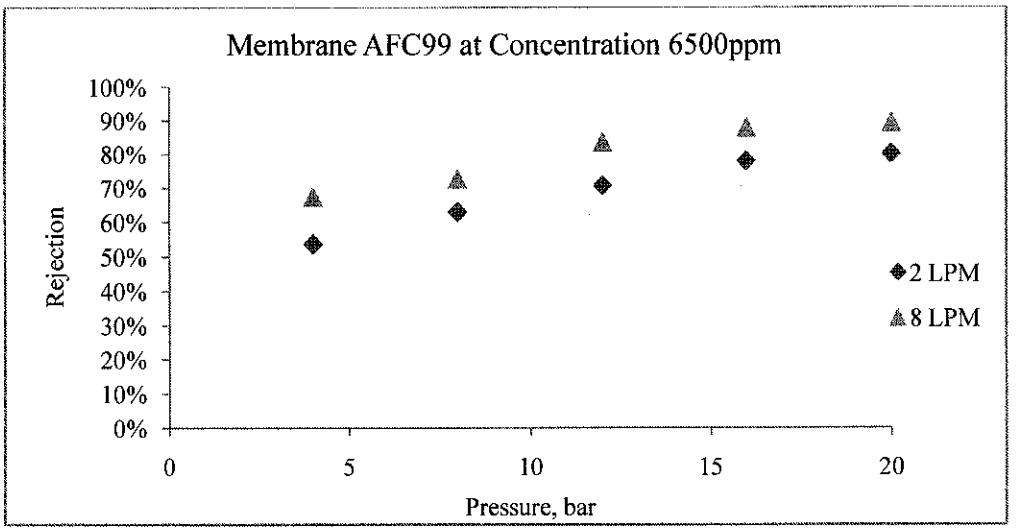

FIGURE 4-16: Rejection vs. Pressure at Constant Concentration 6500ppm for Membrane AFC99

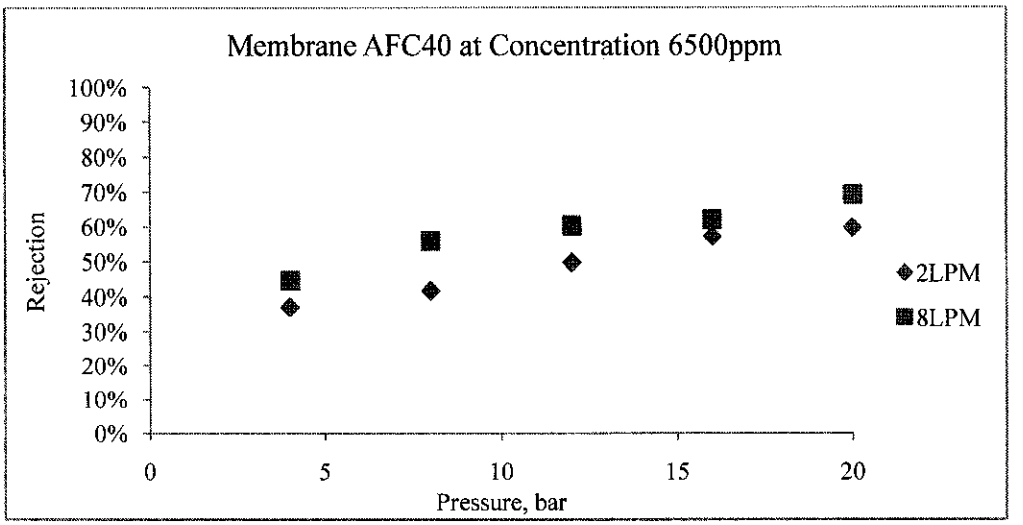

FIGURE 4-17: Rejection vs. Pressure at Constant Concentration 6500ppm for Membrane AFC40

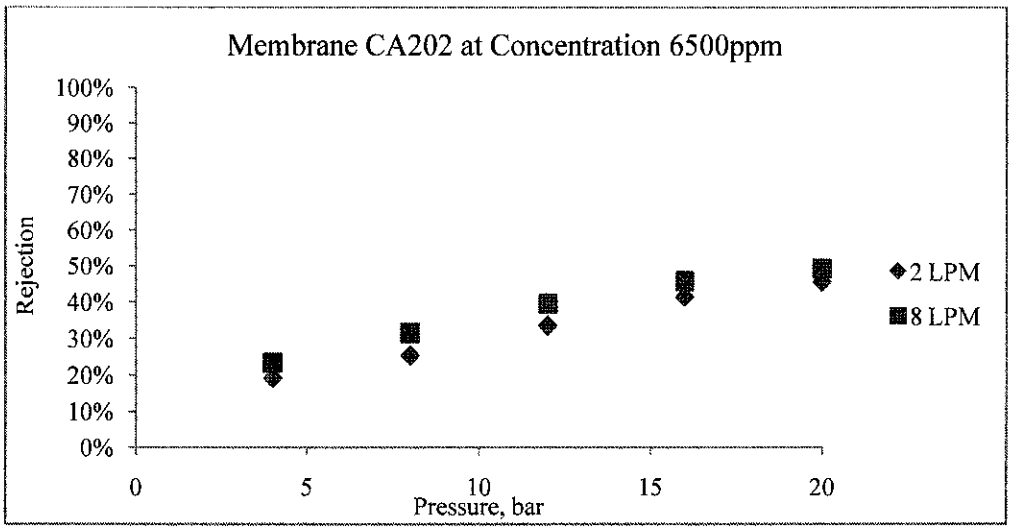

FIGURE 4-18: Rejection vs. Pressure at Constant Concentration 6500ppm for Membrane CA202

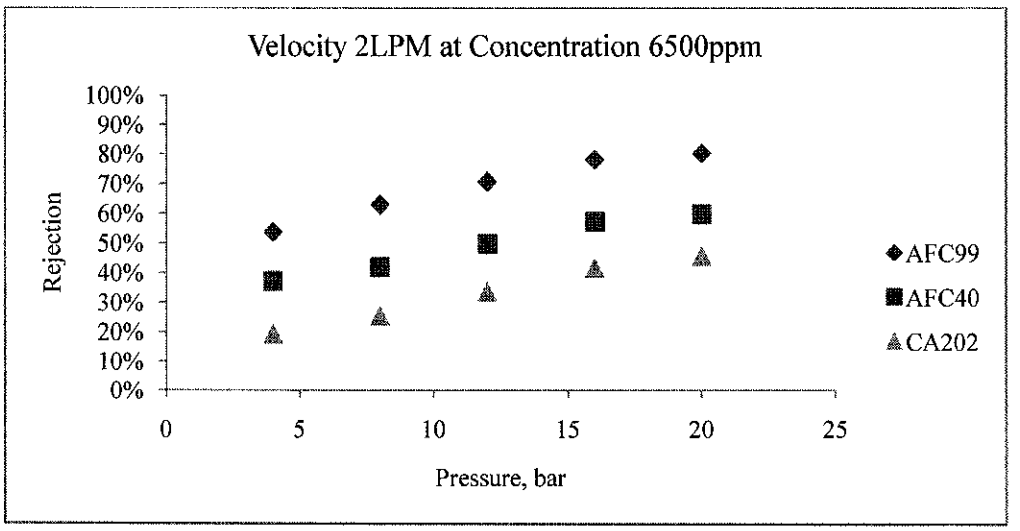

FIGURE 4-19: Rejection vs. Pressure at Constant Concentration 6500ppm and Velocity 2LPM

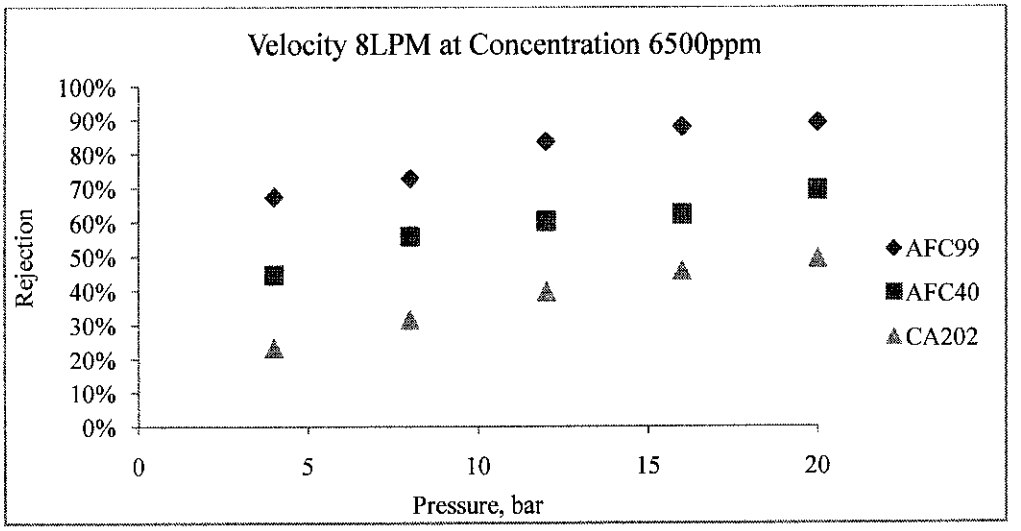

FIGURE 4-20: Rejection vs. Pressure at Constant Concentration 6500ppm and Velocity 8LPM

b) At concentration 52 000ppm

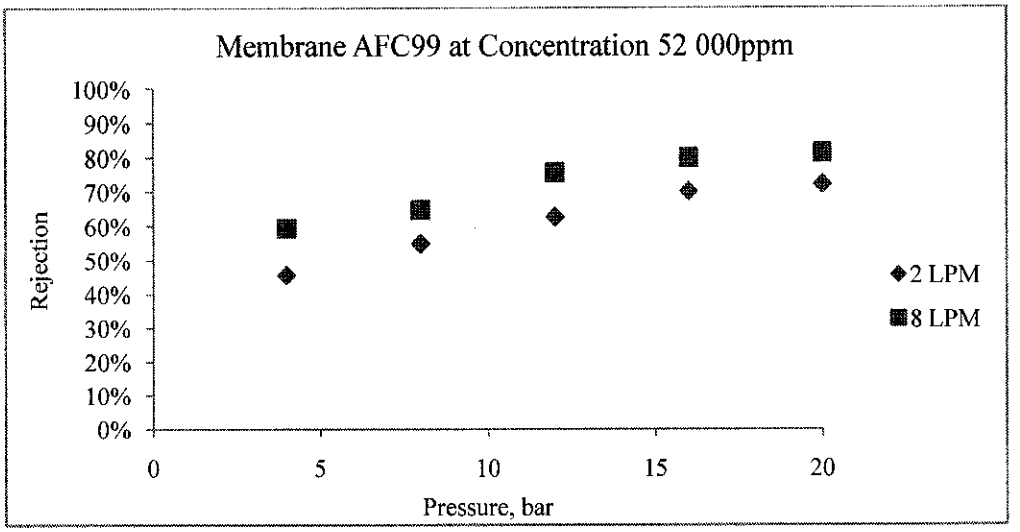

FIGURE 4-21: Rejection vs. Pressure at Constant Concentration 52 000ppm for Membrane AFC99

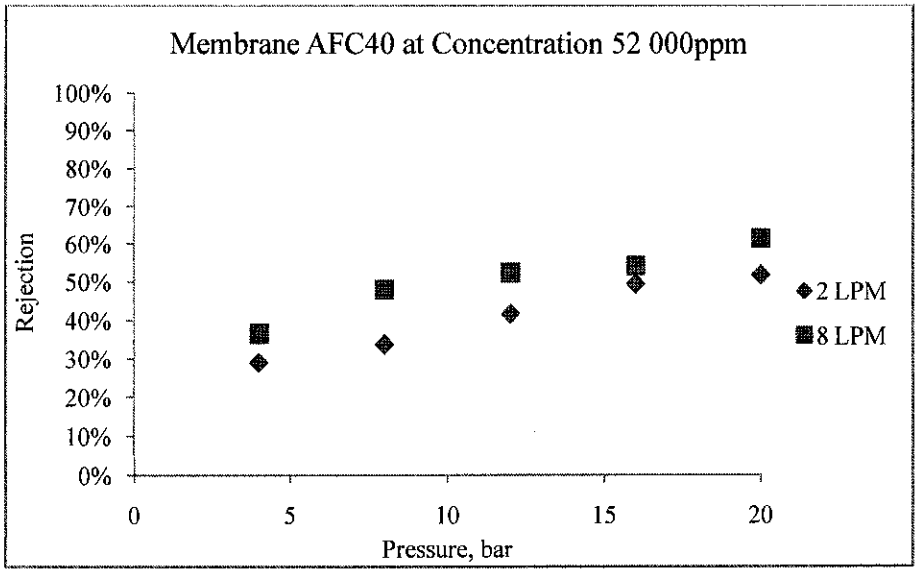

FIGURE 4-22: Rejection vs. Pressure at Constant Concentration 52 000ppm for Membrane AFC40

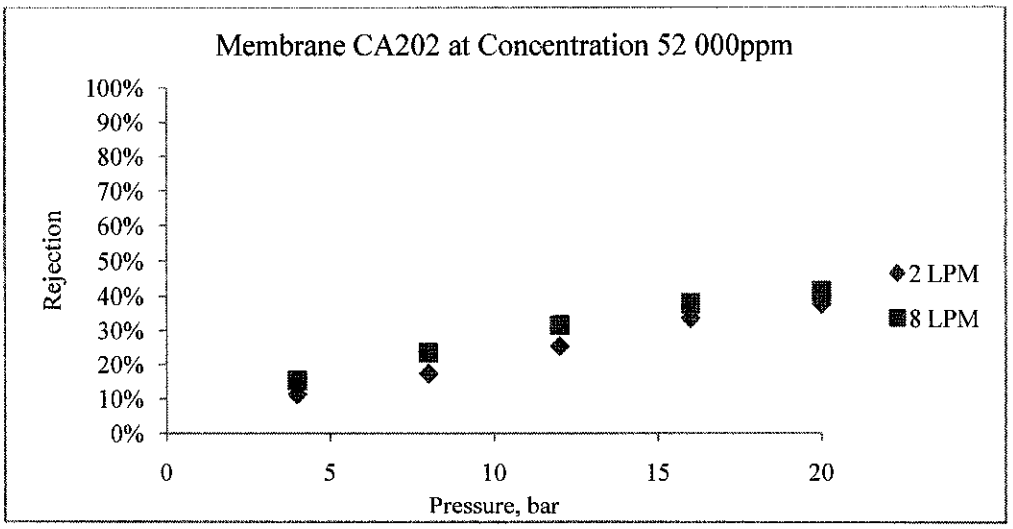

FIGURE 4-23: Rejection vs. Pressure at Constant Concentration 52 000ppm for Membrane CA202

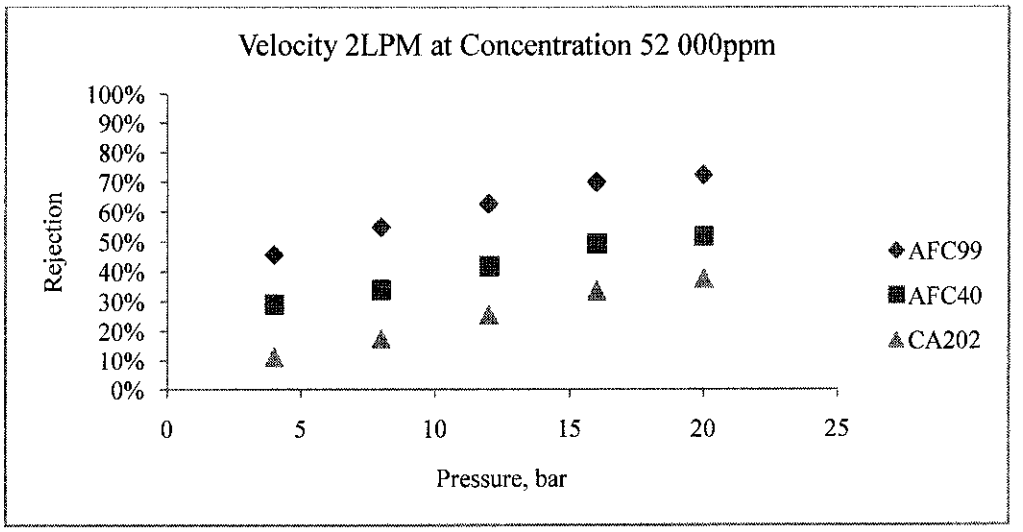

FIGURE 4-24: Rejection vs. Pressure at Constant Concentration 52 000ppm and Velocity 2LPM

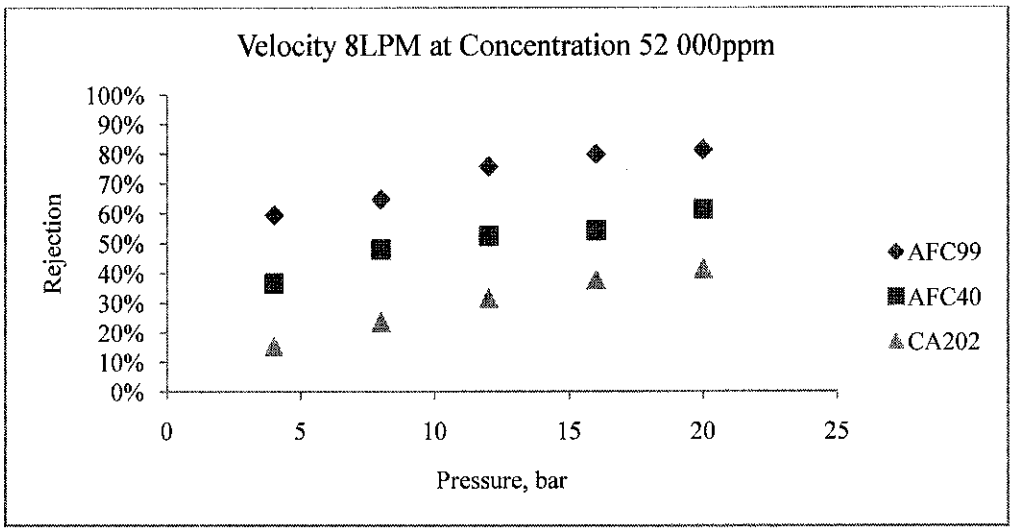

FIGURE 4-25: Rejection vs. Pressure at Constant Concentration 52 000ppm and Velocity 8LPM

Effect of Cross-flow Velocity

Salt rejection was also evaluated at different operating conditions such as operating pressure, feed concentration, cross-flow velocity and types of membrane. For rejection, the concentration was obtained based on calibration curve in APPENDIX G. Rejection increased with increasing pressure (Koyuncu *et al.*, 2003). The highest rejection was obtained for the run conducted at highest operating pressure, 20bar. Result of rejection measurement at different cross-flow velocities and feed concentrations were shown in

FIGURE 4-19, 4-20, 4-24 and 4-25. Amine rejections of the run conducted at high cross-flow velocities were higher than those at low cross-flow velocities due to the higher concentrations on membrane surface. Referring to FIGURE 4-16, for membrane AFC99, 8LPM shows higher rejection level, 89% than 2LPM, 80%. Similarly, membrane AFC40 and CA202 exhibit higher rejection at higher cross-flow velocity as shown in FIGURE 4-17 and 4-18, respectively.

Effect of Feed Concentration

Amine rejection differences decreased as amine concentration increased. Amine rejections for AFC99 were obtained at 80% and 72% for 6500 and 52000ppm of feed concentrations, respectively, for velocity of 2LPM at 20bar. For membrane AFC40 and CA202, the graphs show similar pattern where rejection decreases as feed concentration increases. Comparing between three types of membrane, membrane AFC99 always has highest rejection, followed by AFC40 and CA202. This is because AFC99 has smallest pore size between those three. Also, rejection decreased at higher concentration because of higher concentration polarization and it is related with cross-flow velocity. At low feed concentration, high cross-flow velocity give significant impact to concentration polarization. But at higher concentration, high cross-flow velocity did not give much impact on concentration polarization. The different is not significant because velocity has small influence and the gap between them is quite close. In addition, decrease in amine rejections was observed at high concentrations and low cross-flow velocities as shown in FIGURE 4-19, 4-20, 4-24 and 4-25. This could be explained with reduction in amine hydrophobicity, which results in a thinner concentration polarization layer at high TEA concentrations (Koyuncu *et al.*, 2003). The molecules did not accumulate near membrane surface with the reduction in hydrophobicity at high ionic strengths. However, stagnant gel layer increased and formed as an additional layer on membrane surface for amine rejection at low cross-flow velocities (Koyuncu *et al.*, 2003). This phenomenon increased the effects of cross-flow velocity on amine rejection at low amine content where the rejection differences increased.

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATIONS

From the experiment data, it was observed that parameters are important factors in membrane separation and play important role in the membrane performance. The experimental work shows that the flux of membrane depends on operating pressure, cross-flow velocity, membrane type and feed concentration. The flux increases as the operating pressure increases. Similarly, flux increases as the cross-flow velocity increases. Highest flux was observed for membrane CA202 (104 $L/m^2.h$) at feed concentration 6500ppm, pressure 20bar and cross-flow velocity, 8LPM. Lowest flux was observed for membrane AFC99 (0.5$L/m^2.h$) at feed concentration 52 000ppm, pressure 4bar and cross-flow velocity 2LPM. At high feed concentration, flux decreases and this was due to concentration polarization at the membrane surface. The lower feed concentration would give the better performance, compare to higher concentration which lead to lower flux. The highest flux was membrane CA202, which has highest pore size, followed by AFC40 and AFC99.

It was found that the membrane was able to separate TEA and the separation efficiency depends on operating pressure, cross-flow velocity, membrane type and feed concentration. Membrane AFC99 was found to be best membrane separation, followed by AFC40 and CA202, respectively. The separation efficiency of AFC99 was up to 89%, followed by AFC40 (69%) and CA202 (49%). Membrane AFC99 has tight pore, thus reject highest amine. Rejection increased as operating pressure increases. Similarly, rejection increases as cross-flow increases. But, rejection decreases as feed concentration increases. This is because of high concentration polarization on the membrane surface. The minimum requirement for COD value at discharge point A is less than 100mg/L. At this condition, the requirement can not be satisfied via these membranes. This implies that the efficiency of membrane, AFC99 especially was somewhat lower, which could not bring to required discharge concentration. In term of performance, membrane AFC99 was better than AFC40 and CA202. Further analysis should be conducted in order to achieve required standard.

In term of cost, typically reverse osmosis has a very high price in industry, compared to nanofiltration and ultrafiltration. RO can work as stand alone in separation process which has the highest efficiency among all but the cost of RO will become an issue. The study of amine separation in membrane need to be explored in detailed because the use of membrane is very effective in wastewater treatment plant. The concentration of amines in wastewater need to be reduced, thus reducing the COD value. By revising the result in above section, the membrane separate amine in higher pureness where the feed concentration of amine is low. High concentration may not be suitable to use because of factors of membrane fouling and the parameters used need to be further research. Membrane method is very useful to understand and study the behavior of liquid separation and performance of membrane types.

Few suggestions were also recommended for this project for better observation and evaluation. pH is one of the important factor for molecule permeation through the membrane. At higher pH of the solution, where the molecules are mostly ionized, higher salt rejection will be obtained. The change in rejection behavior is related to the change in configuration of the acid. When the acid or base is in ionized form, the rejection will be higher but in nonionized form, the rejection will be low. So, for this purpose, accurate pH indicator must be included to monitor the pH of the solution.

Membrane material also contributes separation performance. Different materials will require different parameter controls and will exhibit different results for separation performance. Membrane polymer-solute interactions are different in different type of polymer and solute. Expansion studies on this matter should be conducted in future. Therefore, comparison could be made on justifying the most suitable membrane material for amine-water separation through membrane systems. The experiment should be conducted in longer run time because of the effect of membrane compaction, due to different operating pressure and need almost a day to stabilize. Higher operating pressure contributes into higher flux and rejection in separation of contaminant from wastewater until it reaches optimum pressure. Thus, higher range of operating pressure should be put on trial until the separation process reach its optimum operating condition.

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APPENDIX A

PHYSICAL AND CHEMICAL PROPERTIES OF TEA

TABLE A: Physical and Chemical Properties of TEA

Properties	Triethanolamine, TEA
Physical State	Solid/Liquid
Chemical	$(C_2H_5O)N_3$
Molecular Weight, g/mol	149.19
Purity	99%
Appearance	Colorless to pale yellow
Odor	Ammonia
Flash Point -closed cup, °C	179
Flammable Limits; - Lower, %	1.3
- Upper, %	8.5
Autoignition Temperature, °C	315
Vapor Pressure	3.59E-006mmHg (25°C)
Boiling Point, °C	335
Vapor Density (air=1)	5.14
SG, H_20=1, 20°C	1.125
Freezing Point, °C	21
Melting Point, °C	21
Solubility in Water	soluble
pH	10.5 (15g/L H_2O)

APPENDIX B

MEMBRANE INFORMATION

In Membrane Test Unit, below are the equipments consist in it.

Two pumps are provided with the Membrane Test Unit:

Specification	Lowara Centrifugal Pump	CAT Triple Plunger Pump
Max flow rate (LPM)	80	13
Max head	22	-
Max working pressure (bar)	8	7-5
Max liquid temperature (°C)	110	71
Speed (RPM)	2800	1725
Power (HP)	0.5	3.0

TABLE B-2: Membrane Test Unit Pump Specification

A pressure regulator is installed to regulate the operating pressure of the feed system.

Specifications:

Pressure regulated : 7-70 bar

Allowable flowrates : 3.8-38 L/min

1. Tanks and Heating System

   The Membrane Test unit is supplied with a feed tank and a product tank, both having maximum capacity of 15 L. The feed and the product tanks are made of stainless steel for corrosion and chemical resistance. The retentate line is equipped with a unit of shell and tube heat exchanger.

2. Water Flow Meter

   The Membrane Test unit is supplied with a CT Platon water flow meter.

APPENDIX C

SCHEMATIC DIAGRAM OF MEMBRANE TEST UNIT

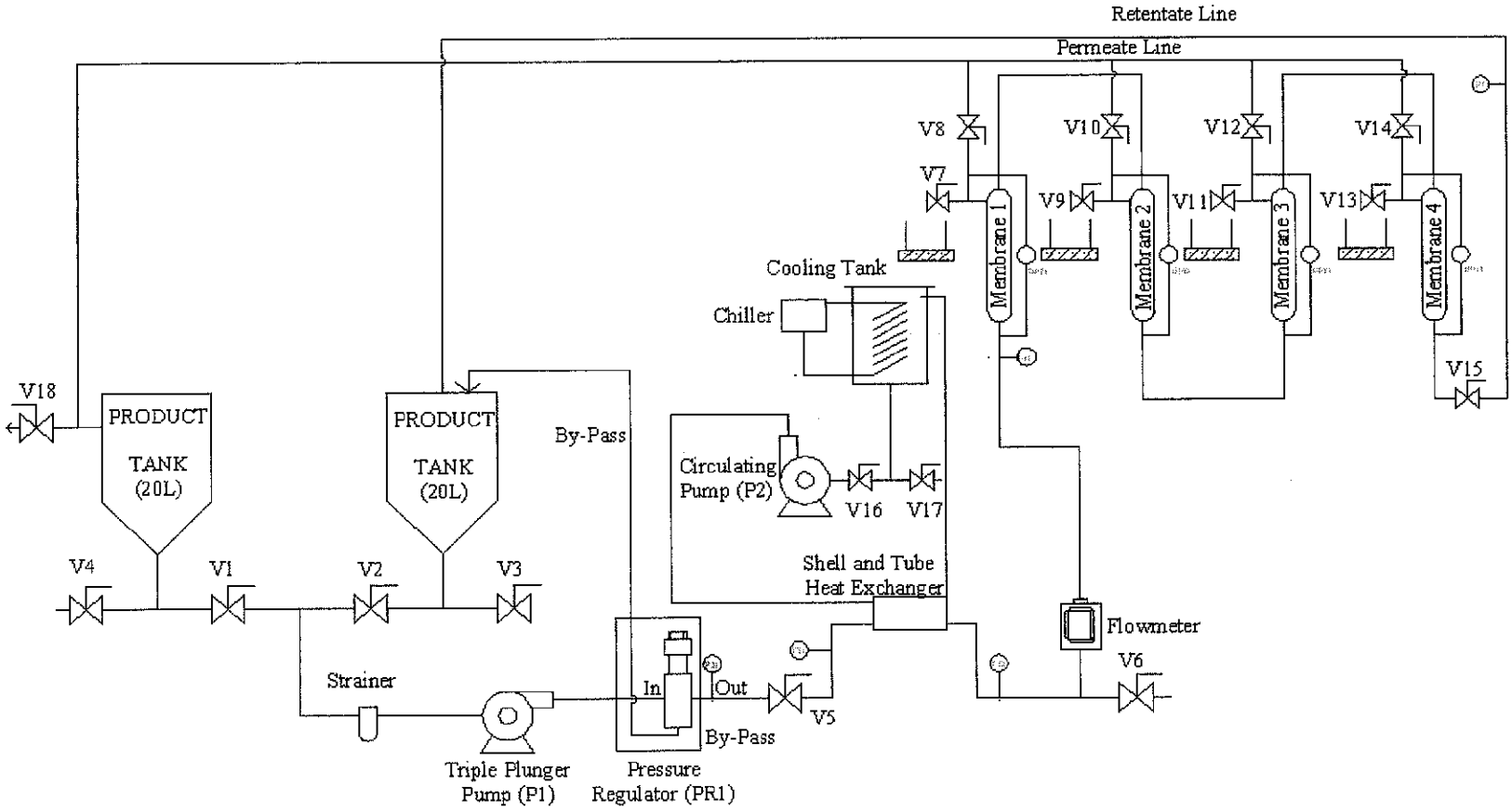

FIGURE C: Process Schematic Diagram

APPENDIX D

COD TEST UNIT

COD Test Unit Start Up

Start-up

1. Place the instrument on a stable, level, heat-resistant surface.
   DANGE! The ventilation slits in the lid must not be covered, or overheating may occur.
2. Plug in the power cord into a power socket (100–230 V +5%/–15%, 50/60 Hz).
3. Switch on the instrument by switching the power switch at the back of the instrument.
4. After initialization, the instrument will beep once, indicating that it is ready for operation. The display always shows the most recent setting of the temperature programs after initialization.

Safety Precautions of COD Test Unit

Use of Hazard Information

If multiple hazards exist, this manual will use the signal word (Danger, Caution, Note) corresponding to the greatest hazard.

DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTE

Information that requires special emphasis.

Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

This symbol, if noted on the instrument, references the instruction manual for operational and/or safety information.

Hot Surfaces. Touching the reactor block surfaces and vials while hot can cause serious burns.

Protective Earth Ground. This product requires a protective earth connection. If not provided with a plug on a cord, connect positive earth to this terminal (U.S. cord set provides ground).

Safety Precautions

Safety Equipment

Use protective clothing when operating the reactor, including goggles or face mask, and gloves.

Reagent Spills

Clean up spilled reagents immediately. If reagent contacts skin, rinse the affected area thoroughly with water. Avoid breathing released vapors. Read the Material Safety Data Sheets (MSDS) supplied with each reagent for complete chemical information.

Fire Hazard

Avoid the presence of flammable liquids near the operating reactor. A fire hazard could be created.

Power Cord

A power cord suitable for 115 V ac line voltage is supplied with the DRB 200.

DANGER! Do not allow the power cord to pass under the instrument.

Specifications

TABLE D: Specification of COD Test Unit

Dimensions	250 x 145 x 310 mm (W x H x D)
Weight	LTV082.53.30001: 2 kg instrument, 3.5 kg with packaging LTV082.53.40001: 2 kg instrument, 3.5 kg with packaging LTV082.53.42001: 2.8 kg instrument, 4.3 kg with packaging
Ambient Operating Temperature	10–45 °C
Storage Temperature	– 40– 60 °C
Relative Humidity	maximum 90% non-condensing
Stored Programs	COD program (150 °C, 120 minutes)
	TOC program (105 °C, 120 minutes)
	100 °C program (100 °C, 30, 60, 120 minutes)
	105 °C program (105 °C, 30, 60, 120 minutes)
	150 °C program (150 °C, 30, 60, 120 minutes)
	165 °C program (165 °C, 30, 60, 120 minutes)
	Easily selected 37–165 °C (no cooling)
Programmable Temperature Range	37–165 °C
Programmable Timer Range	Easily selected 0–480 min; acoustic signal when the set time expires, heating stops when time expires.
Heating Rate	From 20 to 150 °C in 10 minutes
Temperature Stability	± 2 °C
Number of Vials	LTV082.53.30001: 9 holes for 16 vials, 2 holes for 20 mm vials LTV082.53.40001: 15 holes for 16 mm vials LTV082.53.42001: 21 holes for 16 mm vials, 4 holes for 20 mm vials
Power Requirements	100–240 V, +5%/–15%, 50/60 Hz, Protection Class I
Power Input	600 VA
Safety Checks	CE, GS, and cTUVus

APPENDIX E

WATER PERMEABILITY DATA

TABLE E: Water Permeability Data

MEMBRANE		AFC 99				AFC 40				CA 202			
VELOCITY, LPM		2	4	6	8	2	4	6	8	2	4	6	8
PRESSURE, BAR	4	1.0012	1.2425	2.5332	5.2352	5.3317	5.7177	6.5621	7.5513	10.7600	12.5452	15.8866	16.7793
	8	1.1701	2.7865	3.9686	5.9349	8.3474	8.9264	9.3969	10.6996	14.2823	18.2268	19.7829	21.9059
	12	1.3667	5.7057	4.0290	5.4524	11.7129	11.8577	11.7370	12.3764	20.2051	20.5790	20.8685	21.9421
	16	1.4596	1.7732	2.3764	5.5247	16.0314	16.6586	17.3341	17.6840	25.8022	28.1423	29.4451	31.8577
	20	2.6538	4.1375	3.8359	5.8142	18.4439	20.1689	20.0121	20.8082	28.6369	31.0253	30.6996	31.4596

APPENDIX F

TEA SEPARATION DATA

TABLE F: TEA Separation Data of Flux and Rejection

Concentration = 6 500 ppm										
Pressure, bar	4		8		12		16		20	
Velocity, LPM	2									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.8203	0.5374	4.6164	0.6295	6.6598	0.7083	8.0048	0.7809	9.3052	0.8033
AFC 40	5.4789	0.3714	8.9566	0.4185	12.8191	0.4972	11.7720	0.5731	16.3957	0.5973
CA 202	33.3739	0.1928	47.5199	0.2536	55.7201	0.3347	68.8275	0.4156	81.3945	0.4562
Velocity, LPM	4									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.4427	0.5574	4.9397	0.6495	7.0905	0.7283	10.4970	0.8009	11.7346	0.8233
AFC 40	6.5585	0.3914	12.8999	0.4385	15.5802	0.5172	21.0422	0.5931	26.0591	0.6173
CA 202	30.2714	0.2128	45.4198	0.2736	55.0374	0.3547	71.4668	0.4356	84.0796	0.4762
Velocity, LPM	6									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	1.8637	0.5774	5.3366	0.6695	9.8215	0.7483	15.9638	0.8209	20.3402	0.8433
AFC 40	12.5899	0.4114	16.0881	0.4585	23.9288	0.5372	32.6610	0.6131	41.5211	0.6373
CA 202	34.4077	0.2328	46.4282	0.2936	64.8685	0.3747	82.2400	0.4556	99.3631	0.4962
Velocity, LPM	8									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	3.5476	0.6751	7.8637	0.7281	13.3341	0.8370	18.2437	0.8800	23.7780	0.8945
AFC 40	15.0000	0.4463	21.2087	0.5594	27.6261	0.6037	35.6393	0.6220	42.1351	0.6935
CA 202	38.3064	0.2338	51.3727	0.3156	70.3378	0.3957	87.0748	0.4576	104.0060	0.4932

*Flux = $L/m^2.h$

*Rejection = 100%

TABLE F: Cont'..TEA Separation Data of Flux and Rejection

Concentration = 13 000 ppm										
Pressure, bar	4		8		12		16		20	
Velocity, LPM	2									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.0724	0.5074	4.0736	0.5995	5.5609	0.6783	7.2147	0.7509	8.4005	0.7733
AFC 40	3.2678	0.3414	7.2714	0.3885	9.4186	0.4672	11.5899	0.5431	12.9867	0.5673
CA 202	21.6273	0.1628	26.1013	0.2236	51.9131	0.3047	61.1086	0.3856	77.9373	0.4262
Velocity, LPM	4									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	1.7358	0.5274	4.1906	0.6195	6.3353	0.6983	8.4451	0.7709	10.9131	0.7933
AFC 40	3.3932	0.3614	9.8468	0.4085	14.1170	0.4872	19.1098	0.5631	22.4885	0.5873
CA 202	29.0422	0.1828	46.4113	0.2436	59.9759	0.3247	73.5802	0.4056	87.9373	0.4462
Velocity, LPM	6									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	3.9457	0.5474	6.7431	0.6395	11.0820	0.7183	19.2955	0.7909	19.4415	0.8133
AFC 40	6.7949	0.3814	11.0302	0.4285	18.8456	0.5072	32.2171	0.5831	32.6755	0.6073
CA 202	24.9493	0.2028	31.9578	0.2636	55.5959	0.3447	72.1785	0.4256	87.9457	0.4662
Velocity, LPM	8									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	7.3583	0.6451	3.4451	0.6981	5.7720	0.8070	8.0084	0.8500	10.1267	0.8645
AFC 40	8.2027	0.4163	8.8745	0.5294	13.9083	0.5737	19.8577	0.5920	26.4861	0.6635
CA 202	32.5573	0.2038	40.1182	0.2856	59.1616	0.3657	76.9650	0.4276	95.9903	0.4632

*Flux = $L/m^2.h$

*Rejection = 100%

TABLE F: Cont'..TEA Separation Data of Flux and Rejection

Concentration = 26 000ppm										
Pressure, bar	4		8		12		16		20	
Velocity, LPM	2									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	1.5489	0.4874	3.2750	0.5795	4.8902	0.6583	6.0314	0.7309	7.6019	0.7533
AFC 40	0.1918	0.3214	5.3559	0.3685	8.1713	0.4472	9.7274	0.5231	10.5766	0.5473
CA 202	21.2883	0.1428	50.1399	0.2036	61.5163	0.2847	69.3631	0.3656	84.8516	0.4062
Velocity, LPM	4									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	1.6466	0.5074	3.3667	0.5995	5.2823	0.6783	7.0048	0.7509	10.1315	0.7733
AFC 40	2.0977	0.3414	8.0157	0.3885	12.3004	0.4672	14.0639	0.5431	19.1062	0.5673
CA 202	21.5875	0.1628	47.6104	0.2236	62.1339	0.3047	68.2630	0.3856	85.1653	0.4262
Velocity, LPM	6									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.1327	0.5274	4.8094	0.6195	7.8794	0.6983	11.1604	0.7709	15.2557	0.7933
AFC 40	3.4186	0.3614	7.9638	0.4085	12.0374	0.4872	17.2111	0.5631	24.7949	0.5873
CA 202	31.9288	0.1828	34.5718	0.2436	46.5283	0.3247	58.2666	0.4056	81.7370	0.4462
Velocity, LPM	8									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.1508	0.6251	5.0941	0.6781	8.2449	0.7870	16.1846	0.8300	18.3474	0.8445
AFC 40	3.6840	0.3963	7.1351	0.5094	12.0084	0.5537	16.2304	0.5720	24.7720	0.6435
CA 202	34.7165	0.1838	45.4343	0.2656	59.3293	0.3457	66.2244	0.4076	86.4101	0.4432

*Flux = $L/m^2.h$

*Rejection = 100%

TABLE F: Cont'..TEA Separation Data of Flux and Rejection

Concentration = 52 000 ppm										
Pressure, bar	4		8		12		16		20	
Velocity, LPM	2									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	0.5127	0.4574	3.4089	0.5495	3.9493	0.6283	5.1255	0.7009	5.8179	0.7233
AFC 40	1.0724	0.2914	7.5875	0.3385	5.7841	0.4172	10.4029	0.4931	18.0157	0.5173
CA 202	23.8709	0.1128	30.3643	0.1736	33.4403	0.2547	52.8975	0.3356	67.3040	0.3762
Velocity, LPM	4									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	1.6466	0.4774	4.1025	0.5695	4.5151	0.6483	7.2702	0.7209	9.6707	0.7433
AFC 40	1.9855	0.3114	4.9554	0.3585	6.6261	0.4372	11.6273	0.5131	14.7322	0.5373
CA 202	28.3088	0.1328	36.0639	0.1936	41.4837	0.2747	53.4113	0.3556	68.0772	0.3962
Velocity, LPM	6									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.0615	0.4974	4.2316	0.5895	4.9095	0.6683	7.2123	0.7409	9.9168	0.7633
AFC 40	3.2437	0.3314	9.1653	0.3785	10.3583	0.4572	12.0507	0.5331	15.4210	0.5573
CA 202	34.9590	0.1528	50.0265	0.2136	51.6996	0.2947	54.0422	0.3756	68.3317	0.4162
Velocity, LPM	8									
Membrane	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection	Flux	Rejection
AFC 99	2.1990	0.5951	4.5778	0.6481	6.4584	0.7570	7.4101	0.8000	9.9686	0.8145
AFC 40	3.6936	0.3663	9.8770	0.4794	11.1218	0.5237	15.6466	0.5420	16.7201	0.6135
CA 202	37.1821	0.1538	51.2014	0.2356	60.9638	0.3157	64.6755	0.3776	68.8516	0.4132

*Flux = $L/m^2.h$

*Rejection = 100%

APPENDIX G

CALIBRATION CURVE OF TRIETHANOLAMINE, TEA

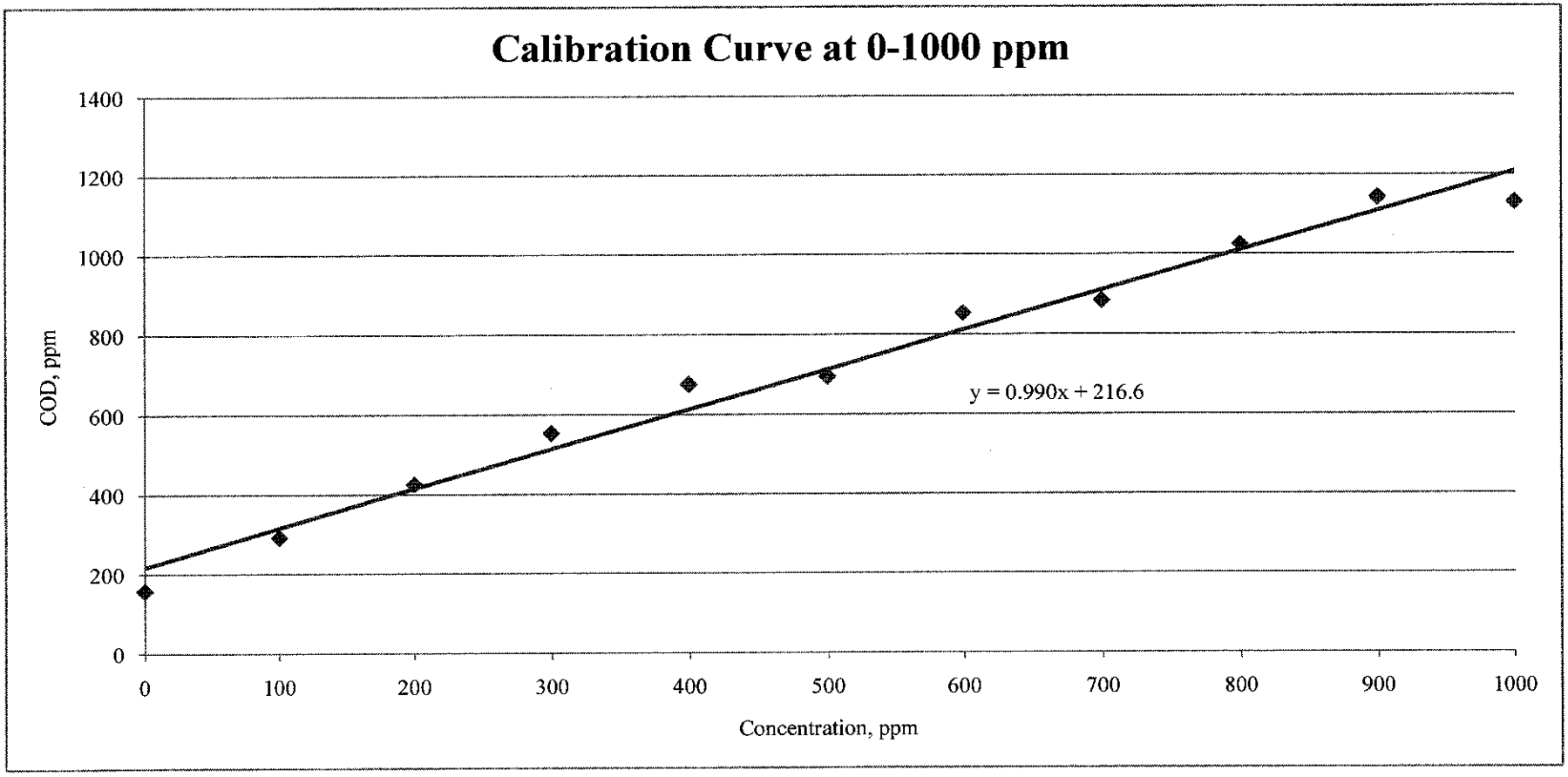

FIGURE G-1: Calibration Curve of TEA at Range 0-1000ppm

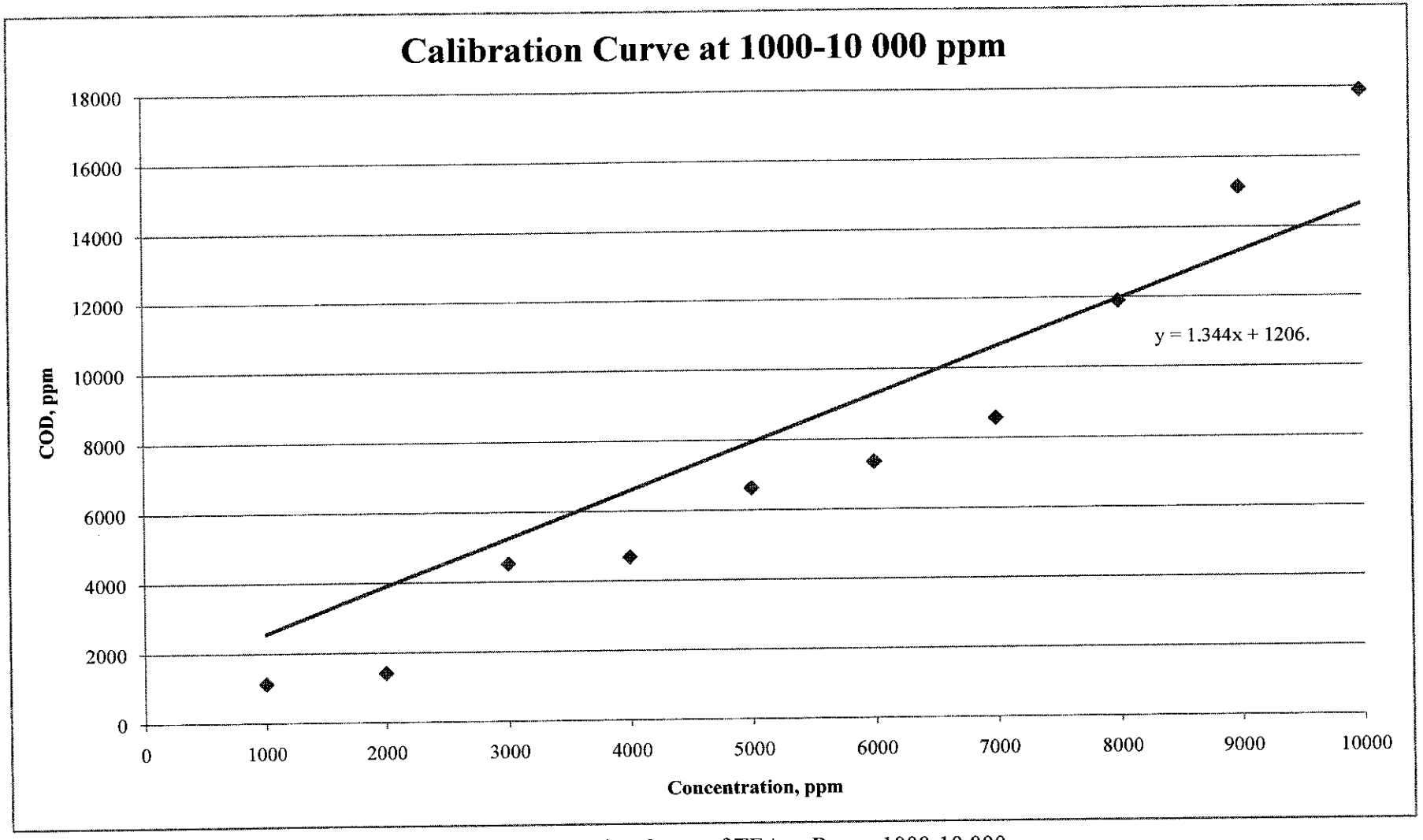

FIGURE G-2: Calibration Curve of TEA at Range 1000-10 000ppm

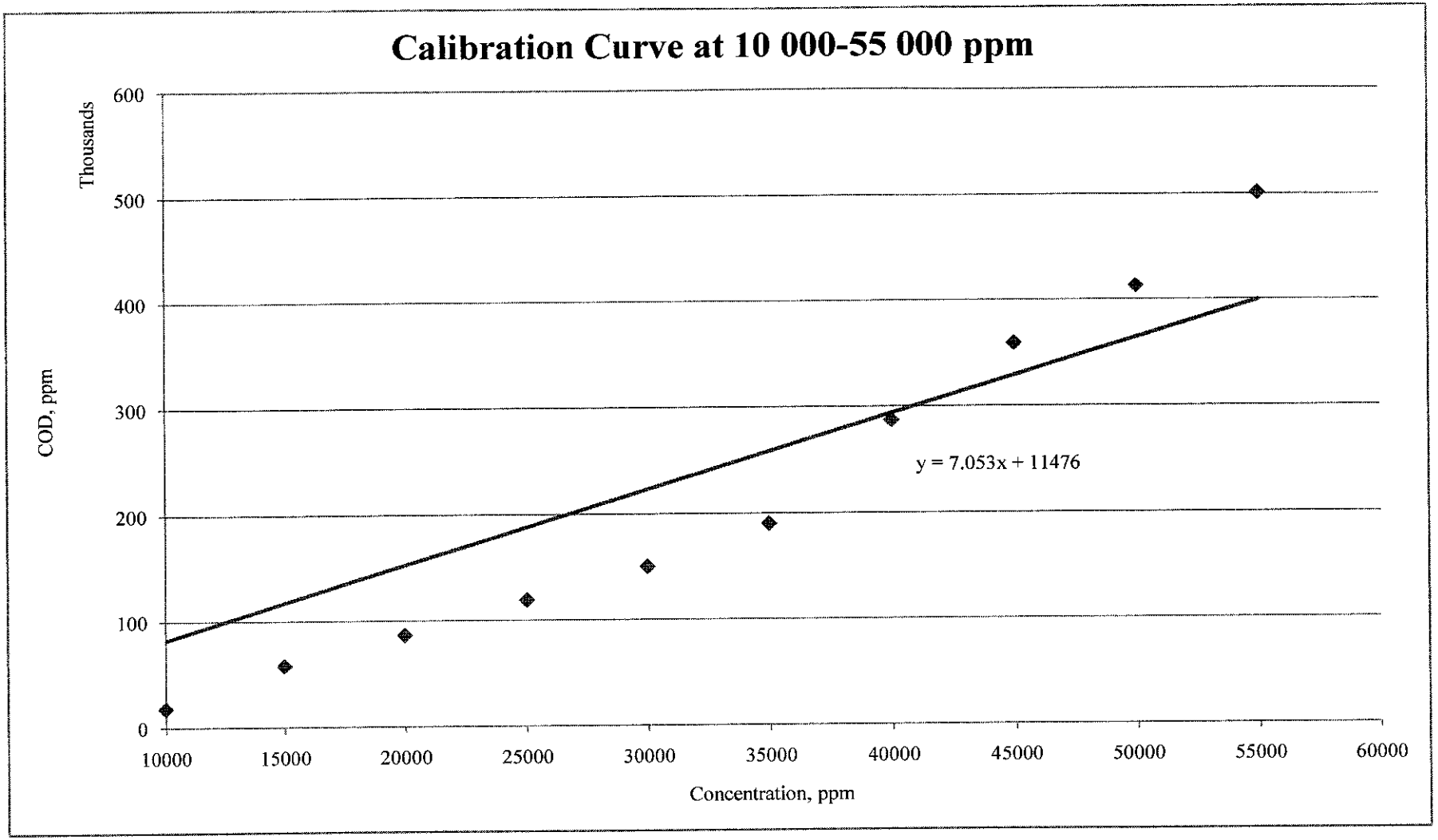

FIGURE G-3: Calibration Curve of TEA at Range 10 000-55 000ppm